

# The Journal of the SOCIETY OF DYERS AND COLOURISTS

Volume 72      Number 7

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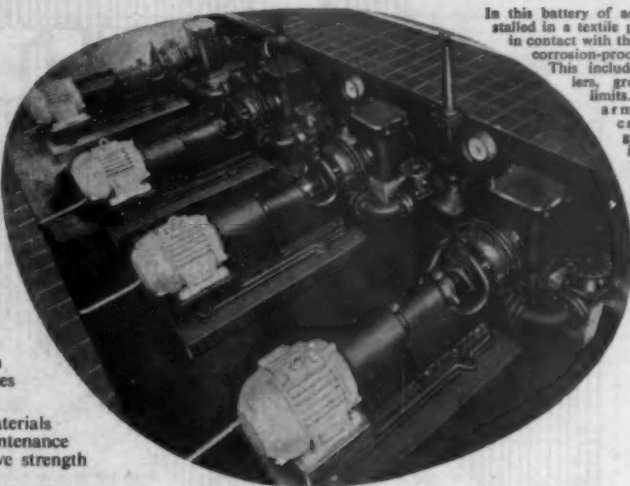
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### Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

#### LECTURES

The Disperse Dyes—their Development and Application

*R. K. Fourness*

Finishing of Fabrics containing Terylene Staple Fibre

*N. M. Mims*

#### F.T.C.C. PUBLICATION

Recommended Conditions for Fading Lamp Operation

*Light Fastness Subcommittee*

#### COMMUNICATIONS

Photochemistry of Dyes on Proteins and other Substrates

*J. W. Cumming, C. H. Giles, and Miss A. E. McEachran*

Influence of Histological Factors on the Attack of  
the Wool Fibre by Various Reagents

*R. L. Elliott and J. B. Roberts*

Photochemical Oxidation of Wool in the  
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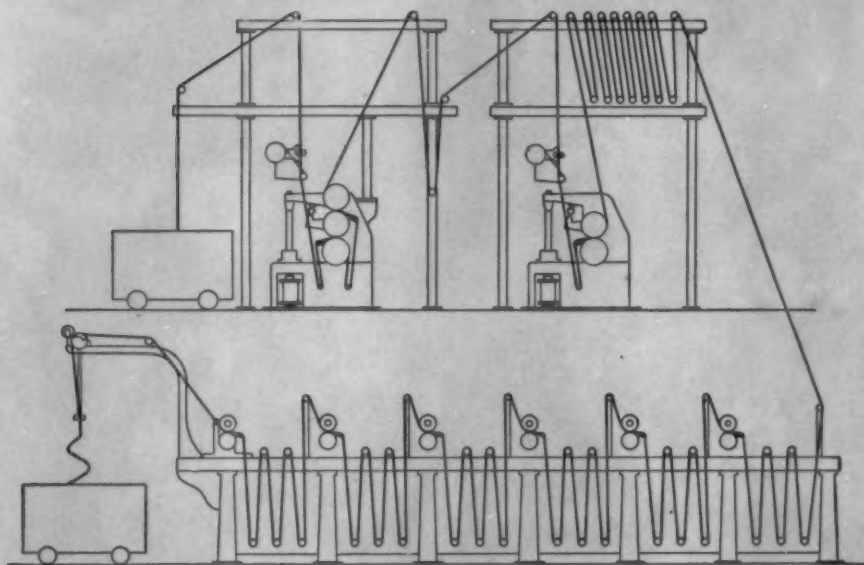
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	VICTORIAN	GEELONG (SUBSECTION)	SOUTH AUSTRALIAN	NEW SOUTH WALES	QUEENSLAND	WEST AUSTRALIAN
Chairman	...	T. F. Hall	J. H. Donaldson	H. D. Bruce	F. Forster	N. Parker
Vice-chairman	...	W. J. Walldie	—	E. Volkuns	H. Senior	H. Knight
Councillor	...	J. Brear	—	—	J. A. Eastwood	—
Honorary Secretary	...	F. Brooke	B. B. Sweeten	N. H. Seager	H. C. Weinberg	G. C. Tune
Honorary Assistant Secretary	...	S. E. Jenkins	—	—	—	D. J. Robb
Honorary Treasurer	...	J. Cooper	G. Goff	J. Allen	F. Powell	J. E. Lee
						C. W. Cruickshank

## DYERS AND COLOURISTS ASSOCIATION OF SOUTH AFRICA

P.O. Box 1700, Durban

Chairman	...	J. H. Allen
Vice-chairman	...	E. Hipperson
Committee	...	E. Cornah
	...	B. Farmer
Treasurer	...	E. Cullis
Secretary	...	G. H. Hayward

## Proceedings of the Society

### Annual General Meeting

The Seventy-second Annual General Meeting of the Society of Dyers and Colourists was held at the Midland Hotel, Manchester, on Friday, 27th April 1956, at 5.0 p.m., the President (Mr. FRED SMITH) being in the chair, and about sixty members of the Society being present.

The minutes of the Seventy-first Annual General Meeting, held on 1st April 1955 at the Queens Hotel, Leeds, were read by the General Secretary (Mr. J. W. NICHOLLS). Mr. W. A. EDWARDS proposed, Mr. H. A. TURNER seconded, and it was agreed that the minutes be accepted as a true record and signed.

The minutes of an Extraordinary General Meeting, held on 27th April 1955 at the Victoria Hotel, Bradford, were read by the General Secretary. Mr. A. G. TYLER proposed, Mr. C. A. MILLS seconded, and it was agreed that the minutes be accepted as a true record and signed.

#### REPORT OF THE COUNCIL

The seventy-second year of the Society has progressed under the leadership of the President, Fred Smith, F.S.D.C., who for over forty-two years has been in office in one form or other serving the interests of the Society. He has devoted himself to the duties of President and Chairman of Council since the death of Frederic Leathley Goodall in June 1954. The President, representing the Society and in his efforts to promote its interests, has travelled over nine thousand miles to take part in meetings and functions in this country and on the Continent.

The Society continues to be in very good heart and emerges from a year of considerably increased activity which has involved the Officers and Committees in a great deal of work. The Honorary Treasurer has reorganised and consolidated the finances of the Society and the Honorary Secretary has introduced new equipment and methods for directing the increasing administrative responsibilities of a Society which is now operating in every country in the world. The Council records grateful appreciation of the very helpful services of these Honorary Officers.

The net increase in membership after allowing for resignations and deaths is 174.

The Annual Dinner, held in Leeds on 1st April 1955, was a great success and was attended by over four hundred diners. The principal guest, whose very entertaining speech will long be remembered, was the late Sir Hubert Houldsworth. The Council records with sorrow his recent sudden death, and extends to Lady Houldsworth and Family deepest sympathy in their bereavement.

The Council announces with very great pleasure the Grant of Arms, Crest, and Supporters by the Three Kings of Arms, who have indicated through the Chester Herald of Arms that they are pleased to recognise the very superior standing of this learned Society. Upon completion, the

Arms, Crest, and Supporters will be displayed on the *Journal* and all literature.

An active part has been taken in the national organisation of the Perkin Centenary Celebrations to be held in London on 7-10th May 1956, and the Council has indicated that all possible encouragement and assistance will be afforded to Sections of the Society who elect to hold local functions in honour of the Centenary. The Council records its thanks to Sir Robert Robinson, O.M., M.A., D.Sc., LL.D., F.R.S., an Honorary Member, who has consented to represent the Society and give the paper on "The Life and Work of Sir William Perkin" at the Perkin Centennial to be held in New York in September 1956.

The Council is deeply appreciative of the service undertaken by Walter Mathers, Managing Director of Lunn & Mathers, Dyers and Finishers, of Farnley, Leeds, who represents the Society as a Trustee of the Perkin Centenary Fund.

The first symposium to be held in Northern Ireland has been an outstanding event of the year, when 387 were present, and the occasion was a happy blend of stimulating discussion and social interests. A lively atmosphere prevailed throughout the sessions with a full exchange of views on current problems of *Bleaching, Dyeing, and Finishing Today* in contribution to the excellent papers given. There was a very welcome attendance of many ladies, who had taken advantage of the facilities for visiting Northern Ireland.

The Guest of Honour and principal speaker at the Symposium Dinner was The Rt. Hon. G. B. Hanna, then Northern Ireland's Minister of Home Affairs, and the Council congratulates and thanks the Symposium Committee for the excellence of the papers and social arrangements. Appreciation is also recorded for the grant of £50 made by the Portrush Urban District Council towards the cost of producing the Symposium brochure.

By the resolution of Council, application was made to rejoin the International Federation of Associations of Textile Chemists and Colourists and this has been unanimously accepted. The President of the International Federation in welcoming the Society has expressed the hope that there will be strengthened the bonds between European textile chemists and colourists and that mutual understanding and co-operation will be fostered. The Society has been invited to send representatives to the next Congress, to be held in Florence on 18th-21st September 1956, when our President is invited to present a paper.

Good relations have been maintained with the affiliated Society of Dyers and Colourists of Australia and the Dyers and Colourists Association of South Africa. In addition, cordial co-operation continues between the Society and the American Association of Textile Chemists and Colorists.

One of the Society's important and most valued functions is the holding of Sectional Meetings, when technical papers followed by discussions attract good attendances. The Council, in congratulating Sections upon these excellent meetings, records its gratitude to the local officers and committees for all their hard work.

Congratulations are extended to the Publications Committee on maintaining the high standard of the *Journal* which has appeared on time throughout the year and Council thanks the authors of papers and communications, abstractors, book reviewers, advertisers, the Indexer, the Editor of Abstracts and Patents, the Editor of the *Journal*, and all who contribute to its success.

The Council wishes to place on record its thanks for donations and is grateful to the Dyers and Finishers Association for the gift of an annual prize of ten guineas for competition between Junior Members, to be allocated to the West Riding Section of the Society.

The Twelfth Mercer Lecture was a great success with 400 present in the Midland Hotel, Bradford, to hear G. Landells, Ph.D., A.R.I.C., F.S.D.C., deliver his address on *Modern Resin Finishing*, and Council is very grateful to the anonymous donor whose financial support of the Mercer Lectures makes these possible.

The continued appreciation of the generous foundation of the George Douglas Lectures by The Bradford Dyers' Association Limited is also recorded.

The work of producing the Second Edition of the *Colour Index* progresses, but the Council, in recognising the difficulties of this task undertaken by the producers, asks that patience will prevail until the *Index* is completed. It is hoped to publish this towards the close of 1956. The Council congratulates and offers encouragement to those who strive to this end.

The Diplomas scheme continues to progress and there is increasing interest in the examinations for the Associateship Diploma.

The Society's Third Annual Golf Competition was held on 8th May 1955 at Meltham, when there were nineteen entrants and appreciation is recorded of the organiser's valuable services, which ensured a very successful and happy occasion.

The Council is very grateful to the firms, colleges, and technical institutions for the help and facilities they have given to the Society.

#### *Reports on Activities of Committees*

**FINANCE COMMITTEE**—This has now been changed to the Finance and General Purposes Committee. During the year the Committee has been especially concerned with the introduction of modern equipment and the reorganisation of internal administration in order to promote efficiency in dealing with the increasing activities of the Society.

**SOCIETY'S MEDALS COMMITTEE**—On the recommendation of the Committee, Council has awarded the Perkin Medal posthumously to the late W. H. Carothers, for the discovery of nylon. This Medal

has also been awarded to John R. Whinfield, C.B.E., M.A., F.R.I.C., for the discovery of Terylene.

A Bar has been awarded to the Gold Medal of P. W. Cunliffe, Ph.D., F.R.I.C., F.T.I., F.S.D.C., for continued valuable services to the Society as Chairman of the Fastness Tests Co-ordinating Committee, and the Society's Silver Medal has been awarded to K. McLaren, B.Sc., F.R.I.C., F.S.D.C., for valuable services to the Society as Honorary Secretary of the Fastness Tests Co-ordinating Committee, and to J. G. Grundy, F.S.D.C., for valuable services to the Society as Chairman of the Washing Fastness Subcommittee, and M. E. Probert, Ph.D., F.R.I.C., for valuable services to the Society as Chairman of the Bleaching Fastness Subcommittee, and also to E. Wilson, B.A., F.S.D.C., for valuable services to the Society as Chairman of the Alkaline Milling, Burnt Gas Fumes, and Perspiration Fastness Subcommittees.

A Diploma expressing the thanks of the Council for services rendered is to be awarded to the other members of the various fastness committees.

The Silver Medal has been awarded to H. W. Ellis, Ph.D., A.R.C.S., A.R.I.C., F.S.D.C., for valuable services to the Society as Honorary Secretary of the London Section from 1938 to 1954, and to J. V. Summersgill, B.Sc., F.S.D.C., for valuable services to the Society as Honorary Secretary of the Publications Committee from 1945 to date.

**WORSHIPFUL COMPANY OF DYERS RESEARCH MEDAL COMMITTEE**—Council report the award of the Research Medal to C. H. Giles, Ph.D., F.R.I.C., F.S.D.C., as the senior author of the papers *A Study of Certain Natural Dyes. I—The Adsorption of Brazilwood and Logwood Colouring Matters by Fibres. II—The Structure of the Metallic Lakes of the Brazilwood and Logwood Colouring Matters*. This award has been approved by the Court of the Worshipful Company of Dyers.

**DIPLOMAS COMMITTEE**—On the recommendation of the Diplomas Committee, Council elected twenty-three Fellows and fourteen Associates. The Diplomas Executive Subcommittee has been dissolved, and an Examinations Board appointed to advise the Diplomas Committee and to act as the executive body to run the examinations.

**PUBLICATIONS COMMITTEE**—The Committee has had an active year and the standard of lectures and communications has been maintained at a high level. The *Journal* is the largest yet published and, with the index, will be almost 1,000 pages. This increase is accounted for partly by the inclusion of the Report of the Fastness Tests Co-ordinating Committee, for which a special format was used, by the publication of the A.S.D.C. Examination Papers for 1954 and 1955 with the examiners' reports, and by a new series of explanatory papers. Council believes all these additions are in the interest of members and welcomes especially the explanatory papers. The series commenced with two papers by H. A. Turner, F.R.I.C., F.T.I., F.S.D.C., on general dyeing theory, and these were followed by an explanation of modern theoretical views on wool dyeing by L. Peters, M.Sc., Ph.D.; further papers are in course of preparation. The

writing of such papers is a difficult task and it is not easy to find authors who have the expert knowledge, are able to write clearly, and can devote sufficient time to such work.

The increase in published matter, together with rising printing costs, has resulted in a substantial increase in *Journal* costs for the year. This position is receiving the close attention of the Finance and General Purposes Committee.

THE TEXTBOOKS AND MONOGRAPHS SUB-COMMITTEE hope to arrange for new publications during 1956. This Committee has very much in mind the needs of candidates for the Associateship.

CLOUR INDEX EDITORIAL PANEL—1955 has been an arduous year for the members of the Editorial Panel. There has been some disappointment because it has not been possible to publish the Second Edition of the *Colour Index* during 1955 as had been anticipated, but everything is being done to complete this task at the earliest possible date, and it was decided to appoint an additional co-ordinator. The Panel gratefully acknowledges that it was possible for C. O. Clark, F.T.I., F.S.D.C., to accept this appointment.

Council appreciates the continued cordial relations with our editorial and production partners, the American Association of Textile Chemists and Colorists, and also thanks the co-operating dye manufacturers for their invaluable assistance and the printers, Messrs. Chorley & Pickersgill Ltd. and Messrs. Percy Lund, Humphries & Co. Ltd., for their co-operation.

FASTNESS TESTS CO-ORDINATING COMMITTEE—There have been four meetings of the Committee during the year with sustained activity on the part of the various subcommittees.

The meeting of the I.S.O. Fastness Subcommittee (TC 38/SC 1) which was held at Scarborough in June 1954 achieved very satisfactory results. Final agreement was reached on twenty-one tests, which will shortly become I.S.O. Recommendations and thus acquire world-wide validity. Fourteen other tests were accepted tentatively and another two were discussed.

The Third Report of the Fastness Tests Co-ordinating Committee was published in the *Journal* in June 1955 and subsequently reprinted under the title *Standard Methods for the Assessment of the Colour Fastness of Textiles*. This report contains all the I.S.O. tests mentioned above, together with a number of others, which, though not accepted internationally, continue to be of importance in this country. Most of the tests are to be published shortly by B.S.I. as British Standards.

A further meeting of ISO TC38/SC1 is to be held in New York in September 1956, when it is hoped that discussions on the fourteen tentative tests will lead to their final acceptance as agreed methods.

The Continental Group (ECE) held a meeting in Brussels in June 1955 at the time of the Exhibition and was attended by the Chairman and other members of the Fastness Tests Co-ordinating Committee.

*Third Report of the Fastness Tests Co-ordinating Committee*—Council thanks and congratulates all concerned with the production of the Report.

PRESERVATION OF HISTORICAL RECORDS COMMITTEE—During the year several gifts of material have been received, the most important being a large collection of dyemakers pattern cards relating to calico printing, for which the Council thanks the donors. Members of the Committee also gave assistance in the preparation of the exhibition of English chintz shown in Manchester in January 1956.

TERMS AND DEFINITIONS COMMITTEE—The Committee has been augmented by the inclusion of additional members. The work has been mainly concerned with reviewing terms and definitions previously considered.

REVIEW OF TEXTILE PROGRESS—This Joint Committee of the Textile Institute and the Society is responsible for the *Review of Textile Progress*, which has maintained its high level of production. Council appreciates the co-operation of this composite body.

#### *Representation on External Bodies*

B.S.I. RAYON INDUSTRY STANDARDS COMMITTEE AND TEXTILE DIVISIONAL COUNCIL—The meetings of the above two committees during 1955, namely the meeting of the Rayon Industry Standards Committee in London on Tuesday, 29th March 1955, and the two meetings of the Textile Divisional Council, held in Manchester on 29th April and 20th September 1955, were attended by the Society's representative. The business was not of any special interest to the Society.

CITY AND GUILDS OF LONDON INSTITUTE—DYING ADVISORY COMMITTEE—There was a substantial increase in the number of entries for all grades of Examinations. A revision of the syllabuses was considered necessary and the new ones will come into operation in the 1956-1957 session. Arising out of a discussion on textbooks it is asked that the Society of Dyers and Colourists notify the City and Guilds from time to time of any of the Society's publications which are considered suitable for students.

REGIONAL ADVISORY COUNCIL FOR TECHNICAL AND OTHER FORMS OF FURTHER EDUCATION FOR MANCHESTER AND DISTRICT—The programme of lectures for the session 1956-1957 includes courses of interest to the Society such as the course on "Physical and Chemical Bases of Fibre Technology" at the College of Technology, Manchester, commencing in the spring of 1956. A series of courses at Salford, Bolton, and Manchester will be held on problems arising from the use of new fibres in blends. The course at Salford will cover weaving problems, that at Bolton spinning problems, and at the College of Technology, Manchester, dyeing and finishing problems. In addition, courses on colour measurement and colour physics are to be organised at the College of Technology, Manchester.

ROYAL TECHNICAL COLLEGE, SALFORD—APPLIED SCIENCES ADVISORY COMMITTEE—The subject of facilities for dyeing is kept alive as an important item in the proposed extensions. Assurances have been given that interest is centred in a strong department, as there is no provision anywhere in the area for part-time study in subjects such as

dyeing, which can cope with day-release students, as distinct from pure chemistry.

**TEXTILE INSTITUTE—TECHNICAL COMMITTEE "D"**—Main items considered were the quantitative separation of fibre blends such as viscose-cotton, and wool blended with nylon, Terylene, cotton, or viscose, and certain methods of analysis are recommended for acceptance as standards for some of these blends. Methods recommended for the removal of added matter from textiles have been published as a British Standard Draft Specification pending its final revision.

**TEXTILE INSTITUTE—TERMS AND DEFINITIONS COMMITTEE**—The main item of interest has been the publication of the second edition of *Textile Terms and Definitions*, which incorporates many of the terms and definitions put forward to the Textile Institute by the Society.

The following Committees report that there has been little or no business of special interest to the Society—

British Biological Stains Commission  
British Standards Institution  
Colour Terminology  
Cotton Industry Committee  
Electric Discharge Lamps  
Linen Industry Standards Committee  
Proofed Clothing—Dyeing and Finishing Processes Committee  
Rotproofing of Textiles other than Clothing Subcommittee and Panel on Standards for Anodic Oxidation Finishes for Aluminium and Aluminium Alloys  
Wash Fastness of Buttons  
Wool Industry Standards Committee  
Royal Society—British National Committee for Chemistry  
Society of Leather Trades Chemists—Fastness Committee  
Textile Institute—Unification of Testing Methods Committee  
Yorkshire Council for Further Education—County Advisory Committee for Textiles

The services of those representing the interests of the Society on all Committees are very much appreciated and Council concludes this report with thanks to the staff of the Society for their greatly valued contribution in its service throughout the year.

The Honorary Secretary (Mr. L. MORTON WOOD), in moving the adoption of the Annual Report, paid tribute to the late Mr. H. Jennison for his help and encouragement, and for his many years of service to the Society as Honorary Treasurer and in other ways. He referred also to the untiring efforts of Mr. Fred Smith as President during a very busy time. He wished to thank all concerned in the work of the Society—individuals, colleges, firms, etc.—and to express appreciation of the time and energy devoted to the Society.

The Colour Index Editorial Panel was to be congratulated on having the Second Edition of the *Colour Index* ready for publication by the autumn of 1956, and the success of the diplomas scheme was

shown by the fact that 42 candidates had entered for the A.S.D.C. examination in 1956. The Mercer Lecture and the Golf Competition had been very successful, and he was pleased to report that the Kings of Arms had granted a coat of arms and supporters to the Society. The Northern Ireland Symposium had been a great success, naturally at some cost to the Society.

The Perkin Centenary celebrations would be held within the next two weeks, and there was still time to register for the various functions.

Finally, he wished to express his thanks to the staff of the Society and especially to Mr. Nicholls.

Mr. J. W. REIDY, in seconding the adoption of the Report, paid tribute to the work of the late Mr. H. Jennison as Chairman of the Finance Committee.

The motion was carried.

#### ANNUAL ACCOUNTS

The Honorary Secretary (Mr. L. MORTON WOOD), in presenting the accounts for the year ended 31st December 1955, said that he had had to undertake this task at rather short notice in view of the recent death of the Honorary Treasurer. Most items were very level from 1954 to 1955, but wages and salaries showed some increase. The increased cost of publication of the *Journal* reflected its increased size in 1955, due largely to special publications, such as the fastness tests. As acting Honorary Treasurer he had very carefully gone through the accounts, and he felt sure that they gave a sound and true picture of the state of the Society's finances.

Mr. A. P. KERSHAW seconded the adoption of the Balance Sheet and Accounts, and the motion was carried.

#### AMENDMENTS TO THE BYE-LAWS

The Honorary Secretary moved in accordance with the recommendations of Council the re-inclusion of the following Rules in the Bye-laws of the Society—

##### *Management of Finance*

1. The management of the finance of the Society shall be vested in a Finance and General Purposes Committee, consisting of the Honorary Treasurer, Honorary Secretary, Honorary Deputy Treasurer, and not less than three other members of Council, who shall conduct the general financial affairs of the Society.

##### *Appointment of Trustee of Property and Investments*

2. (a) The Council shall have power by resolution to appoint a bank or other trust corporation (hereinafter called the Corporate Trustee) to be Trustee of all or any of the property and investments of the Society on such terms as to remuneration as may be agreed upon between the Corporate Trustee and the Society, and all such property and investments of the Society shall be vested in or transferred to the Corporate Trustee when appointed. A copy of such resolution purporting to be signed by the Chairman of the meeting at which the same was passed, together with one other member of the Council who was present at such meeting, shall be sufficient evidence for all purposes of the due appointment of the Corporate Trustee.

(b) All property and investments so vested in, or transferred to the Corporate Trustee shall be dealt with both as to capital and income in such manner as from time to time may be directed by resolution of the Council. A copy of every such resolution purporting to be signed by the Chairman of the meeting at which the same was passed, together with one other member of the

## Balance Sheet as at 31 December 1955

31.12.54				31.12.54				ASSETS			
SURPLUS AND LIABILITIES								Approx. Middle Market Value		At Cost	
Accumulated Funds								31.12.54			
Account								£ s. d.		£ s. d.	
At				At							
31.12.54				31.12.54							
33,690											
Accumulated Funds at											
1,155	...	...	37,068 1 5								
Add—Library											
Introduced as Asset											
Net Surplus for the year											
1,861				25,329							
100				1,861							
3,438	37,068			100							
				2,000							
1,861	Foundation Fund	...	1,850 13 0	2,000	George Douglas						
100	Knecht Memorial Fund		100 0 0	2,000	Lecture Fund...	...	2,000 0 0				
	George Douglas			2,000	George Douglas						
2,000	Lecture Fund...	...	2,000 0 0	2,000	Fund Investments	...	1,400 12 2			2,000 0 0	
200	George Douglas										
2,209	Accumulated Income	206 4 8	2,206 4 8								

## Consolidated General Revenue Account—continued

31.12.54	EXPENDITURE	£ s. d.	£ s. d.	31.12.54	INCOME	£ s. d.	£ s. d.
3,021	Brought forward ...		4,439 8 2	30,198	Brought forward ...		20,661 15 0
-	Diploma Costs ...		118 10 10				
803	Sectional Costs ...		865 17 1				
	Depreciation of Office Furniture and Equipment ...		85 14 0				
33	Mercer Lecture ...		11 8 0				
47	Journal Stock—1st January 1955	631 0 1					
560	Printing and Publishing of Journal ...	9,863 14 3					
7,643	Postage—Journal Circulation, etc. ...	616 2 2		166	Donations ...		171 0 0
629	Salaries, Wages and Honoraria ...	2,210 0 0					
2,166	Repurchase of Society's Journals ...	60 4 3					
21	Abstractors' Fees and Book Reviews ...	340 15 9					
402	Miscellaneous Publications ...	- - -					
26	Publication Committee Expenses ...	83 19 1					
67	Repairs ...	- - -					
31	Rent, Rates and Insurance ...	170 10 0					
168	Electricity and Heating	27 4 10					
40	Depreciation of Office Furniture and Equipment ...	49 3 7					
34	Telephone ...	51 10 0					
43	Travelling and Hotel Charges ...	66 2 9					
167	Printing and Stationery	125 0 0					
104	Incidental Office Costs	85 0 0	14,318 6 0				
103	Annual Meeting and Dinner Meeting ...	81 0 1					
63	Dinner ...	- - -	81 0 1	325	Excess Income from Sales of Publications		68 19 5
128	Donations and Subscriptions ...		34 7 0				
37	Unpaid Accounts						
	Provisions and amounts written off ...		124 4 9				
744	Library Upkeep ...		23 2 2				
13	Colour Index (2nd Edition)						
	Expenses incurred but not allowable per agreement with A.A.T.C.O. ...		91 14 11				
96	Fastness Tests Account						
514	Stock 1st January 1955	333 15 0		83	Grey Scales ...	166 7 1	
3	Materials Purchased ...	70 4 3		19	Staining Scales ...	26 0 1	
5	Postages ...	4 3 9		17	Reports ...	124 2 0	
80	Loose Patterns and Bunches ...	8 5 0		234	Materials ...	267 19 0	
	Committee Costs—Hire of Meeting Rooms, Travelling, etc. ...	15 14 2		283	Bunches and Loose Patterns ...	668 17 2	
51	Reports ...	102 18 2		334	Stock—31st December 1955 ...	418 6 3	1,678 12 7
653			625 0 4				
17,824			20,818 14 7				
	Balance—Being Surplus for the Twelve Months carried down ...		1,746 12 11				
3,316							
£1,630			£22,565 7 6	£1,630			£22,565 7 6
	Extraordinary Expenditure	£ s. d.	£ s. d.		Balance—Being Surplus for the Twelve Months brought down ...		1,746 12 11
328	Scarborough Conference	- - -	- - -	3,316			
	Excess Expenditure over Receipts concerning Northern Ireland Symposium ...		337 6 1				
-	Historical Records Committee Expenditure		48 19 10				
-	Special Service Accountancy Charges in respect of the years: 1953-1954 ...	115 0 0					
50	1955 ...	120 15 0	235 15 0				
	Net Surplus for the Twelve Months ...		1,124 12 0				
3,438							
£3,816			£1,746 12 11	£3,816			£1,746 12 11

## COLOUR INDEX (SECOND EDITION) ACCOUNT

	£ s. d.	£ s. d.	31.12.54	£ s. d.
31.12.54				
4,669 Stock of Paper 1.1.55 ...		4,668 10 0	4,669	4,668 10 0
2,975 Work in Progress 1.1.55 ...		1,047 0 0	1,047	2,770 0 0
Expenses				
Wages and National Health Insurance ...	1,092 0 0		138	Investment Income
4,132 Printing and Publishing ...	2,708 0 0		7,267	Midland Bank Ltd. Deposit Account ...
221 Advertising and Postages ...	292 2 6			Balance carried to Colour Index
35 Telephone ...	35 10 6			Accumulations Account ...
15 Electricity and Heating ...	17 10 0			
16 Storage and Incidental				
Office Costs ...	27 15 8			
Cleaning ...	50 0 0			
175 Rent, Rates and Insurance ...	203 1 8			
30 Audit and Accountancy ...	30 0 0			
7 Bank Charges ...	6 4 9			
43 5,474 Repairs and Renewals ...	41 3 1	4,508 8 9		
£13,118		£10,218 18 0	£13,118	£10,218 18 0

## COLOUR INDEX (SECOND EDITION) ACCUMULATIONS ACCOUNT

	£ s. d.		£ s. d.
7,266 Accumulated Total at 1.1.55 ...	14,552 10 6	14,553	Accumulated Total at 31.12.55 ...
Balance brought forward being Excess of			
Expenditure over Income for the year ...	2,623 11 0		
7,267			
£14,553	£17,176 7 6	£14,553	£17,176 7 6

## GEORGE DOUGLAS ACCUMULATION FUND ACCOUNT

	£ s. d.		£ s. d.
Lecture Expenses ...	- - -	181	Balance per Accumulated Income Account
Bank Charges ...	2 0 0		31.12.54 ...
Balance per Accumulated Income Account			Investment Income (Gross) ...
31.12.55 ...	266 4 8	60	3% British Transport Stock 1978/88 ...
£211	£268 4 8	£211	

## SCHEDULE OF DEVELOPMENT ACCOUNT INVESTMENTS

	Balance 1954	Alterations	Balance 1955	Middle Market Value 1954	Middle Market Value 1955	Middle Market Quotation
£2,703 3s. 8d. 3% British Transport Guaranteed Stock 1978/88 ...	2,598 17 0		2,598 17 0	2,417 15 7	2,034 7 10	78½
£750 2½% Funding Stock 1956/61 ...	750 0 0		750 0 0	749 1 3	675 0 0	90
£546 3½% Conversion Loan ...	557 0 0		557 0 0	482 10 7	407 9 1	74½
£255 2½% Defence Bonds ...	255 0 0		255 0 0	255 0 0	255 0 0	—
£2,749 16s. 0d. 3% Savings Bonds 1960/70 ...	2,750 0 0		2,750 0 0	2,631 4 4	2,213 11 10	80½
£750 2½% Exchequer Stock 1955 ...	750 0 0		750 0 0	750 4 8	615 0 0	82
500 National Savings Certificates 15s. Units 7th Issue ...	375 0 0	Converted into 3% Funding Stock 1966/68	375 0 0	375 0 0	375 0 0	—
£2,000 3% British Gas Stock 1990/95 ...	2,000 0 0		2,000 0 0	1,750 0 0	1,430 0 0	71½
£2,017 11s. 6d. 3% Treasury Stock 1966 or after ...	1,937 1 0		1,937 1 0	1,663 12 5	1,311 8 0	65
£901 3s. 6d. 3% Southern Rhodesian 1971/73 ...	910 13 9		910 13 9	315 11 2	689 8 0	76½
York County Savings Bank ...	4,080 17 10	Interest credited during the year £107 11s. 5d	4,196 3 3	4,080 17 10	4,196 3 3	—
£1,500 12s. 9d. 3% British Electricity Guaranteed Stock 1968/73 ...	1,495 19 0	Defence Bonds Interest credited during the year £7 14s. 0d.	1,495 19 0	1,418 2 5	1,183 12 7	78½
Post Office Savings Bank ...	1,049 14 8	Interest credited during the year £26 4s. 6d.	1,075 19 2	1,049 14 8	1,075 19 2	—
£1,000 Chesterfield Corporation 3% Stock 1960 ...	993 15 0	Interest credited during the year £83 17s. 0d.	993 15 0	1,000 0 0	900 0 0	90
Midland Bank Ltd. Deposit Account ...	3,678 4 9		3,662 1 9	3,678 4 9	3,662 1 9	—
£1,037 17s. 3d. Treasury 3½% Stock 1970/81 ...	1,007 3 4		1,007 3 4	1,025 13 6	844 11 3	81½
£220 3½% Defence Bonds ...	220 0 0		220 0 0	220 0 0	220 0 0	—
	£25,320 8 4		£25,554 13 3	£24,762 12 2	£22,088 13 3	

## SCHEDULE OF FOUNDATION FUND ACCOUNT INVESTMENTS

	£ s. d.		£ s. d.	£ s. d.	£ s. d.
£1,245 2½% Defence Bonds ...	1,245 0 0		1,245 0 0	1,245 0 0	1,245 0 0
1,000 National Savings Certificates 10s. Units 8th Issue ...	500 0 0		500 0 0	500 0 0	500 0 0
£68 8s. 6d. 3% Savings Bonds 1960/70 ...	69 9 0		69 9 0	65 12 11	55 0 2
£37 9s. 3d. Treasury 3½% Stock 1970/81 ...	36 4 0		36 4 0	35 15 2	30 12 6
	£1,850 13 0		£1,850 13 0	£1,846 8 1	£1,830 12 8

## GEORGE DOUGLAS LECTURE FUND

£1,988 17s. 3% British Transport Stock 1978/88 ...	£2,000 0 0	£2,000 0 0	£1,735 17 4	£1,460 12 2	78½
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## KNECHT MEMORIAL FUND

£98 16s. 6d. 3% Southern Rhodesia 1971/73 ...	£100 0 0	£100 0 0	£89 8 9	£75 12 0	76½
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Council who was present at such meeting, shall be sufficient authority for the Corporate Trustee to act thereon and the Corporate Trustee shall be free from all responsibility for all acts and things done in conformity with such resolution.

(c) The Corporate Trustee shall only be responsible for the property or investments belonging to the Society actually transferred to or vested in the Corporate Trustee and the Corporate Trustee shall not without its consent in writing be required to accept or hold any leasehold or other property or investments which may render the holder thereof liable for the performance of any covenants or for the payment of any money.

#### *Savings Banks*

In view of Bye-law No. 42 stipulating that sums not exceeding in the aggregate £5,000 by deposit in Savings Banks for a period not extending beyond the thirty-first day of December 1955 the Council by Resolution recommends the following amendment to this Bye-law—

3. That Bye-law No. 42 be amended to extend the authority for deposits in Savings Banks for a period as may be determined by Resolution of Council which shall also determine the sums in aggregate so deposited.

The Honorary Secretary explained that Council on the advice of the Society's bankers had resolved to recommend the re-inclusion in the Bye-laws of those Rules governing the appointment of a corporate body as trustee holders of the Society's investments.

With regard to Council's resolution No. 3, this requirement was due to Bye-law No. 42 stipulating that sums not exceeding an aggregate of £5,000 could be deposited in Savings Banks only for a period terminating 31st December 1955.

Mr. R. J. HANNAY seconded.

Mr. H. FOSTER proposed that there be added to the paragraph under the heading of "Management of Finance" the following words—

They shall be elected by and responsible to Council.

Mr. J. BARRITT supported because he felt it desirable that it be made clear that control remained vested in Council.

Sir ROBERT ROBINSON suggested the following wording—

The management of the finance of the Society shall be vested in a Finance and General Purposes Committee, which shall be appointed by and responsible to the Council and shall consist of the Honorary Treasurer, the Honorary Secretary, and not less than three other Council members elected by the Council.

With this agreed alteration the Chairman declared the Resolutions numbered 1, 2, and 3 carried.

#### *Trustee of the Perkin Fund to be Ex-officio Member of Council*

The Resolution of Council which recommended that the Society's representative Trustee of the Perkin Centenary Fund be an *ex-officio* member of the Council of the Society was moved by Mr. A. W. CARPENTER, seconded by Miss E. SMITH, and unanimously agreed to for inclusion in the Bye-laws of the Society.

#### *Past Presidents to be Members of Council*

Mr. G. G. HOPKINSON proposed and Mr. R. K. FOURNESS seconded that in accordance with Council's recommendation the Immediate Past President and his two predecessors automatically become members of Council, with the one longest

out of the presidency retiring and being replaced by the Immediate Past President upon the election of a President-elect.

Mr. H. A. BRASSARD enquired as to their title and would they be known as "Past Presidents" or as "members of Council".

The President explained that the intention was to retain the three last retiring Presidents' experienced services as *ex-officio* members of Council.

Mr. C. O. CLARK thought that this would be weakening Council members directly elected by the members.

Mr. HANNAY explained how the proposal would operate, but on a show of hands the proposal was declared lost.

The President said that Council would reconsider the proposal and resubmit it to an Extraordinary General Meeting if necessary.

#### *Honorary Deputy Treasurer*

It was agreed this appointment was not now necessary, and the resolution was accordingly deleted from the Agenda.

#### *Awards of Medals and Diplomas*

Mr. C. O. CLARK moved, Dr. A. F. KERTESS seconded, and it was agreed that the following be incorporated in the Bye-laws of the Society—

That the Council is authorised to recommend and award Medals, Diplomas, and to elect Fellows and Associates of the Society.

#### *ELECTION OF PRESIDENT*

Mr. FRED SMITH, in proposing Mr. CLIFFORD PAINE for election as President, said that, unlike himself a dye user, Mr. Paine was a dyemaker and had all the qualifications necessary for the position, which he was sure he would hold with dignity and never let the Society down.

Mr. G. S. J. WHITE, in seconding the motion, said that Mr. Paine would be an altogether worthy representative of the Society, both at home and abroad.

The motion was carried with acclamation.

Mr. CLIFFORD PAINE said that it was difficult to express his gratitude for the honour. He was very conscious of the distinguished men who had filled the office, and he could not hope to attain their level. It would be a privilege and a responsibility to represent the Society at public functions, and he hoped that he would do it with credit and distinction. His immediate predecessor had performed a difficult duty in difficult circumstances arising from the sudden death of Mr. F. L. Goodall: many schemes were half developed and many problems half solved. And Mr. Fred Smith had put tremendous work into his presidential duties. We owed him a great debt of gratitude.

#### *ELECTION OF VICE-PRESIDENTS*

The General Secretary announced the results of the postal ballot as follows—

Professor W. Bradley	558 votes
Mr. W. Penn	576 votes

The President then declared Mr. W. Penn elected Vice-president to serve for six years and Professor W. Bradley elected Vice-president to serve for three

years (to fill a casual vacancy resulting from the resignation of Mr. Fred Smith as Vice-president).

#### ELECTION OF ORDINARY MEMBERS OF COUNCIL

The General Secretary announced the results of the postal ballot for four Ordinary Members of Council as follows—

Mr. C. O. Clark	488 votes
Dr. A. Datyner	388 votes
Mr. R. K. Fourness	493 votes
Mr. W. L. Langton	425 votes
Mr. C. P. Tattersfield	407 votes

The President then declared Messrs. C. O. Clark, R. K. Fourness, W. L. Langton, and C. P. Tattersfield elected Ordinary Members of Council.

#### ELECTION OF AN HONORARY TREASURER

It was agreed that Council fill the vacancy of Honorary Treasurer in accordance with the requirements of the Bye-laws.

#### RE-ELECTION OF HONORARY SECRETARY

The President moved, the Immediate Past President seconded, and it was unanimously agreed

that Mr. L. Morton Wood be re-elected Honorary Secretary.

Mr. Wood suitably replied.

#### APPOINTMENT OF AUDITORS

Mr. L. M. Wood proposed, Mr. J. BARRITT seconded, and it was unanimously agreed that Messrs. Rawlinson, Greaves & Mitchell, Chartered Accountants, of Bradford, Yorkshire, be re-appointed Auditors.

#### THANKS TO SCRUTINEERS

The President recorded thanks to the ballot scrutineers, Mr. R. Grice and Mr. A. Waddington.

#### VOTE OF THANKS TO RETIRING VICE-PRESIDENT AND ORDINARY MEMBERS OF COUNCIL

On the motion of Mr. G. G. HOPKINSON, seconded by Mr. R. K. FOURNESS, a vote of thanks was accorded the retiring members of Council (*Vice-president*—Mr. W. A. Edwards; *Ordinary Members of Council*—Messrs. D. A. Derrett-Smith, N. Hamer, A. P. Kershaw, and J. V. Summersgill).

## Annual Dinner

The Seventy-second Annual Dinner of the Society was held at the Midland Hotel, Manchester, on Friday evening, 27th April 1956, under the presidency of Mr. Clifford Paine.

The principal guests were The Right Honourable G. B. Hanna, Q.C., M.P. (*Minister of Finance in the Government of Northern Ireland*) and Alderman R. S. Harper, J.P. (*Deputy Mayor of Manchester*). Among other guests were Mr. E. I. Noble (*Honorary General Secretary of the Society of Dyers and Colourists of Australia*), Dr. D. W. Kent-Jones (*President of the Royal Institute of Chemistry*), Mr. J. R. Ruck-Keene (*General Secretary of the Chemical Society*), Mr. K. W. Luckhurst (*Secretary of the Royal Society of Arts*), Sir Robert Robinson (*Chairman of the Perkin Centenary and Honorary Member of the Society*), Mr. Arthur H. Brewin (*Prime Warden of the Worshipful Company of Dyers*), Mr. E. B. Laycock (*Master of the Worshipful Company of Feltmakers*), Mr. G. F. Williams (*Chairman of the Association of British Chemical Manufacturers*), Mr. H. Golden (*Secretary of the Dyers and Finishers Association*), Dr. B. V. Bowden (*Principal of the Manchester College of Technology*), Professor R. H. Peters (*Professor of Textile Chemistry in the Manchester College of Technology*), Professor G. Gee (*Professor of Physical Chemistry in the University of Manchester*), Mr. J. Wilson (*Director of Research of the British Rayon Research Association*), Mr. C. G. Hulsoe (*Secretary of the Dyestuffs Control, Board of Trade*), Mr. D. W. B. Scott (*President-elect of the Bradford Textile Society*), Mr. W. Hiles (*Deputy President of the Bradford Engineering Society*), Mr. L. E. Morris (*Editor of "The Dyer"*), Mr. R. D. Chorley, Mr. H. Clayton, Mr. J. W. F. Morton, Mr. L. Savage, Herr W. Stauffacher, Mr. L. E. Jones (*Honorary Patent Agent*), Mr. J. R. Whinfield (*Perkin Medallist*),

Dr. P. W. Cunliffe and Mr. G. S. J. White (*Gold Medallists*), Dr. C. H. Giles (*Worshipful Company of Dyers Research Medallist*), Dr. H. W. Ellis, Mr. J. G. Grundy, Mr. K. McLaren, Dr. M. E. Probert, Mr. J. V. Summersgill, and Mr. E. Wilson (*Silver Medallists*), and Mr. J. Boulton (*Chairman of the Manchester Section*).

The following officers and other leading members of the Society also were present—Mr. Fred Smith (*Immediate Past President*); Mr. H. H. Bowen, Mr. F. Scholefield, and Dr. C. M. Whittaker (*Past Presidents*); Professor W. Bradley, Mr. R. J. Hannay, Mr. G. G. Hopkinson, Mr. W. Penn, and Mr. H. A. Turner (*Vice-presidents*); Mr. C. O. Clark, Mr. A. S. Cluley, Mr. A. S. Fern, Mr. R. K. Fourness, Mr. H. Hampson, Mr. J. W. Reidy, Mr. C. P. Tattersfield, Mr. A. G. Tyler, and Dr. T. Vickerstaff (*Ordinary Members of Council*); Mr. F. Attack, Mr. A. W. Carpenter, Mr. C. W. Green, Mr. H. R. Hadfield, Mr. C. A. Mills, Mr. J. Porter, Mr. R. G. J. Reid, Mr. H. W. Taylor, and Mr. H. Turner (*ex-officio Members of Council*); Mr. K. Meldrum and Mr. N. G. Morton (*Junior Branch Officers*); Mr. J. Barritt (*Chairman of the Publications Committee*); and Mr. L. M. Wood (*Honorary Secretary*).

The total number of diners was 520, a record in the history of the Society.

#### WELCOME TO MANCHESTER

After the toast of "Her Majesty the Queen", the Society was welcomed to Manchester by—

Alderman R. S. HARPER, J.P. (*Deputy Mayor of Manchester*), who said that, according to the timetable, he had finished speaking thirty-five minutes earlier. He would not make a long speech, which might jeopardise the time allocated to other activities. In conveying the hearty greetings of the City of Manchester, he would like to congratulate

the Manchester Section of the Society on its Diamond Jubilee and to say how much the work of the Society was appreciated in Manchester. He much regretted that the Lord Mayor could not be present, but since the latter was at that moment in Chicago, that was physically impossible. Finally, he felt sure that his example would enable his hearers to enjoy subsequent speakers all the more. (*Acclamation and cries of "encore".*)

"THE SOCIETY OF DYERS AND COLOURISTS"

The Right Honourable G. B. HANNA, Q.C., M.P. (*Minister of Finance in the Government of Northern Ireland*), said that he had been honoured by the invitation to propose the toast of the Society, and memories of the enjoyable evening he had spent at Portrush had made him accept with alacrity: the Society had paid his little country a compliment in deciding to hold its 1955 symposium there. Judged by press reports during the past ten days, the English reputation for warm hospitality was dwindling, but personally he did not feel unduly concerned, as he had made no speeches in India.

He wondered why he had been invited to propose this toast. As a cabinet minister he could make a highly colourful speech with purple patches, but one cannot sell a threadbare garment however highly coloured it may be. He apologised for the fact that his designation on the menu card as "Minister of Home Affairs" had been made out of date by recent changes in the Northern Ireland Government—he had migrated from prisons to pensions, from capital punishment to capital investment.

He would like to offer his hearty congratulations to Mr. Clifford Paine on the latter's election as President of the Society. Mr. Paine had had great success in his profession: he had been sent from Yorkshire to Lancashire to make some colour, of which the world was in dire need. The new President was a keen cricket fan, and had purchased a television set so that he might see something of the game.

The Society was concerned with fixing colour, and he was concerned with fixing (but not politically!) wages and prices, in both cases with the aim of achieving stability. Everyone knew how to solve the problem of inflation, but few were ready to make the necessary sacrifices. We were producing more than before the 1939–1945 war, but paying ourselves a great deal more. Members of the Society had a vital part to play in overcoming the technical difficulties associated with the increase in productivity which was required. The Industrial Revolution had resulted from the inventive genius of our people, and it should not be beyond our abilities to break the present vicious circle of rising prices and rising wages. Northern Ireland was only a small part of the United Kingdom, but its export industries made a relatively large contribution to the balance of trade.

He would like to congratulate Mr. J. R. Whinfield on the award of the Perkin Medal, for the discovery of Terylene polyester fibre. In ten days' time would be celebrated the centenary of Perkin's famous discovery of Mauve, whose successful development owed much to Professor A. W. Hofmann's kindness

in not being jealous of his pupil's success and to the support of Perkin's father and the dyeing firm of Pullars. Similarly, with more understanding in youth, more adventure in age, and more determination in us all, we would be able to overcome our present difficulties. He had become serious because he felt that members of the Society had a tremendous contribution to make to the peace and the prosperity of this country. He therefore had very great pleasure in proposing the toast: "The Society of Dyers and Colourists".

Mr. CLIFFORD PAINE, B.Sc. (*President of the Society*), said that he had been looking forward to hearing Mr. Hanna speak, and he had not been disappointed. He was not sure whether the Minister's recent change of job was a translation or a promotion—whether he should commiserate with or congratulate—but the delicate relation between home affairs and finance was known to all. They were grateful for a speech full of wit and wisdom.

Every member of the Society received inspiration from the past achievements of the industry. The elegant elaboration of classical organic chemistry and the development of technology had been applied to the adornment of the human race, and had led to great social changes, not always foreseen. He would like to adopt a motto from Professor Waller of Manchester: *Cherish the past, adorn the present, and create for the future*. He had been encouraged on his visits to the Continent and to America by the high esteem in which the Society's *Journal* was held abroad. The Society would receive added lustre from its work on the Second Edition of the *Colour Index*, undertaken jointly with our American colleagues of the American Association of Textile Chemists and Colorists. Members of the Society must not be content to be scientists and technologists, but must be also men of affairs in industry.

An important object of the Society was to encourage education, and he would like to extend good wishes to Dr. B. V. Bowden in his work of increasing the scope and the value of the Manchester College of Technology. He wished also to pay a personal tribute to the Corporation of Manchester for relinquishing parental control of the College and thus enabling it to expand, for the problem of technical education goes to the heart of our prosperity.

The Perkin Centenary Fund had been initiated and now stood at about £40,000. There was still scope for further contributions. No doubt the Society would be able to offer useful advice to the trustees of the Fund on the uses to which it could be put.

In recent weeks he had taken the opportunity of looking at the textbooks available to modern students, and had been disturbed at the large proportion of American books. Many of these appeared to him to be deficient in their presentation of the historical background of the subject—a matter of considerable educational importance.

The Manchester Section was celebrating sixty years of vigorous activity, with the early years of which Dr. E. Knecht, of the College of Technology,

had been closely associated. The Research Medal of the Worshipful Company of Dyers had been awarded a total of 38 times, of which 13 had been to members of the Manchester Section. In view of his (the speaker's) racial origins from across the Pennines, he could not be accused of local bias.

Finally, Mr. Paine said that he had the interests of the Society at heart. In the words of Shakespeare's sonnet, he would say:

My nature is subdued to what it works in, like the dyer's hand.

#### "OUR GUESTS"

Mr. JOHN BOULTON, M.Sc.Tech., F.R.I.C., F.T.I., F.S.D.C. (*Chairman of the Manchester Section*) said that the unusually distinguished list of guests did the Society great honour.

With his bleaching and dyeing ancestors, his Orange outlook, and his taking silk in 1941, the Rt. Hon. G. B. Hanna had a close connection with our industry. He had the peculiar distinction, as recently as 1953, of being returned unopposed on the first occasion in Northern Ireland when a seat had not been contested.

This Society has always had within its membership a satisfying number of scientists distinguished in the academic field. Although it is not every Annual Dinner that has entertained a Past President of the Royal Society, it was on the cards that sooner or later Sir Robert Robinson would dine with us. Two distinctions, however, have made it imperative that this year he should. Firstly, Council has conferred upon Sir Robert the highest honour which we have—our Honorary Membership. Secondly, in this, the Perkin Centenary year, Sir Robert is pre-eminent as the Chairman of the Centenary Celebrations. In a gathering largely of chemists, to describe the distinctions and achievements of Sir Robert would be supererogatory; it would also take a long time. What we would gain from such an exercise, however, would be a highly coloured view, both broad and deep, of those developments in the science of organic chemistry that have enlivened most of the past half-century. I need only mention how much the chemistry of alkaloids owes to his work, as does our understanding of the chemical nature of penicillin, and that in two broad and widely differing fields—those of biosynthesis and of the electronic theory of organic reactions—his work has helped in a unique way to create an epoch.

It must have been noticeable to citizens less fortunate than we how many great men of the present era, no less than of other times, have passed through the University of Manchester. The Emeritus Waynflete Professor of Chemistry in Oxford arrived there, as several of his fellows have done, via the Chair of Organic Chemistry at Owen's College, which he occupied as far back as 1922–1928. Edgar Noble, whom—and not merely in passing—we welcome as the Honorary General Secretary of the Society of Dyers and Colourists of Australia, will no doubt remember with us that the first university chair to be adorned by Sir Robert was that of Pure and Applied Organic Chemistry in the University of Sydney.

I am told that two of Sir Robert's recreations

have been climbing and music. He will know of the proximity of this city to classic rocks and fells, and of its great musical traditions—and the following extract from the letter of an ex-student, newly in his first job, written to a parent, might assure him of our state of grace:

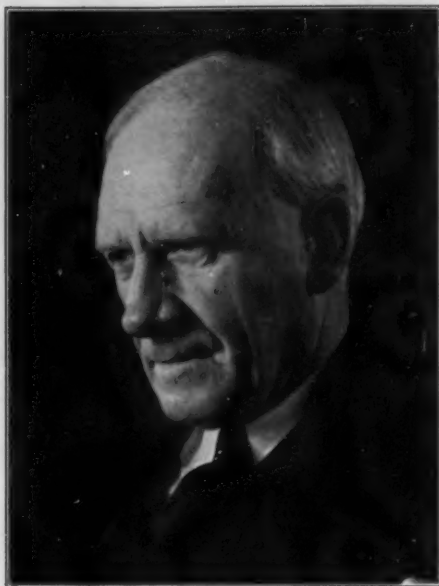
Here in Manchester the principal and old established interests appear to be cricket and textiles—in that order. The younger men in our laboratory, however, have wider interests and talk of only three things, the other two of which are climbing and the Hallé Orchestra.

In the current number of *Endeavour* is a brilliant article by Sir Robert on the Perkin family of organic chemists, wherein a remarkable family tree has been constructed. Sir Robert, in compiling a time scale of great chemists since Perkin Senior, has dealt all too modestly with his own connections.

The Society's Perkin medal has been awarded, on some few occasions, for work in fibre science. To the names of Chardonnet, Henri Dreyfus, and C. F. Cross as recipients of the Perkin medal, we are now adding those of W. H. Carothers and J. R. Whinfield. John Whinfield is here with us tonight and requires no introduction to this company. I think we all take an unusual pleasure in the fact that the great discovery which we honour tonight was made in a dyehouse laboratory, and whether he likes it or not, we had already claimed John Whinfield as one of us. I have said that it is not unusual that distinguished scientists should be our guests, but John Whinfield is unusual in being a member—and a great one—of that very rare class, the scientist inventor.

In the third London Lecture, which appeared recently in our *Journal*, was an account of the ancient guilds of the City of London. None who read this paper can fail to appreciate how right we are to honour our great city companies. A week ago I had the great pleasure of dining with Mr. A. H. Brewin and saw him present to Charles Giles the 38th award of the Worshipful Company of Dyers Gold Medal for research in the field of dyeing. It is our custom to welcome the Prime Warden of the Worshipful Company at our Annual Dinner and it is with especial pleasure that we receive Mr. Brewin, newly elected to that high office, and Mr. D. Balfour Park. With no less pleasure do we welcome Mr. E. B. Laycock, the distinguished Master of the Feltmakers Company. I shall hope that his presence here might prompt some worker in the field to aspire to an equal distinction with our Dyers' medallists in gaining the so far elusive award which the Society is proud to be concerned with, the Research Medal of the Worshipful Company of Feltmakers.

During recent months the Manchester College of Technology has obtained its own charter. It had long housed the University Faculty of Technology, and its degrees in technological sciences have over many years been carried into every branch of industry all over the world. But now it is an authority in its own right, and you will all wish to congratulate the Principal, Dr. B. V. Bowden, on the status to which the College, early in his reign as its dean, has attained. As far as we in the industry are concerned, the principal jobs of the College of Technology are to teach, to encourage research, and



Studio Edmund

Professor Sir ROBERT ROBINSON  
O.M., D.Sc., LL.D., F.R.S., Nobel Laureate

Honorary Membership in recognition of invaluable  
services to the Society and outstanding researches  
on colouring matters



JOHN REX WHINFIELD  
C.B.E., M.A., F.R.I.C., F.T.I.(Hon.)

Perkin Medal for the discovery of Terylene Polyester  
Fibre



PERCY WALMSLEY CUNLIFFE  
Ph.D., F.R.I.C., F.T.I., F.S.D.C.

Bar to the Gold Medal for continued valuable  
services to the Society as Chairman of the Fastness  
Tests Co-ordinating Committee



CHARLES HUGH GILES  
Ph.D., F.R.I.C., F.S.D.C.

Worshipful Company of Dyers Research Medal for  
1954-1955 as senior author of the papers: *A Study  
of Certain Natural Dyes—I and II*



HOWARD WINDLEY ELLIS  
Ph.D., A.R.C.S., A.R.I.C., F.S.D.C.

Silver Medal for valuable services to the Society as Honorary Secretary of the London Section from 1938 to 1954



JAMES GIBSON GRUNDY  
F.S.D.C.

Silver Medal for valuable services to the Society as Chairman of the Washing Fastness Subcommittee



KEITH McLAREN  
B.Sc., F.R.I.C., F.S.D.C.

Silver Medal for valuable services to the Society as Honorary Secretary of the Fastness Tests Co-ordinating Committee



MAURICE ERNEST PROBERT  
Ph.D., F.R.I.C.

Silver Medal for valuable services to the Society as Chairman of the Bleaching Fastness Subcommittee



JOHN VICKERS SUMMERSGILL  
B.Sc., F.S.D.C.

Silver Medal for valuable services to the Society as Honorary Secretary of the Publications Committee from 1945 to date



EDMUND WILSON  
B.A., F.S.D.C.

Silver Medal for valuable services to the Society as Chairman of the Alkaline Milling, Burnt Gas Fumes, and Perspiration Fastness Subcommittees

ultimately to provide our industry with the technologists which are so vitally needed. Anyone who has read the press on this subject of the training of scientists and technologists will know the calibre of its new Principal. That is especially true for those of us outside the faculty. Those inside know it more urgently. As an old student of the College and a frequent visitor, I have the impression that Principal Bowden does not confine his salutary activities to the press, and that things are moving very rapidly during the present process of massive extension going on under his charge.

Part of that process has been the establishment of Manchester's first Chair of Textile Chemistry, and a guest we are all delighted to have with us is its first occupant, Professor R. H. Peters.

The University of Manchester is here with us in the distinguished presence of Professor G. Gee, who was appointed to the Chair of Physical Chemistry some few years ago. We must congratulate him now on his appointment during the present session as the Samuel Hall Professor of Chemistry. Incidentally, any old Mancunians who are here know that this is a position of singular distinction.

This seems to be a time of celebrations: of centenaries and jubilees. This year the firm of Morton Sundour celebrated its golden jubilee, and we are proud and happy to have with us Mr. Jocelyn W. F. Morton, the chairman of that splendid undertaking. Last night I came across some words very recently written by Mr. Morton:

We still may not always be able to give the exact hue that either our artists or our customers may want, as there are still some unobtainable in the necessary fastness, and we *refuse* to put in colours that will disappear before the effective life of the fabric is over.

I quote this because it deals beautifully with two major fallacies of modern life and commerce—that the artist's inspiration is sacred beyond the interference of mundane science, and that the customer is always right.

Amongst other of our many guests from industry are Mr. H. Clayton, Managing Director of the Geigy Co. Ltd., and Herr W. Stauffacher, Director of Sandoz Ltd. of Basle. With the latter I should like to couple in welcome *all* our visitors from overseas.

As the dyeing industry has a turnover of £100 million per annum, it is surprising to overseas visitors to find that it has no research association of its own. Perhaps this is not necessary in view of the existence of the research associations devoted to cotton, wool, and rayon. We welcome the Director of Research of the British Rayon Research Association, Mr. John Wilson.

Amongst guests for whom there is an especial welcome are those from other learned societies. Sitting by me here is Dr. D. W. Kent-Jones, the distinguished President of the Royal Institute of Chemistry. And we have Mr. J. R. Ruck-Keene, General Secretary of the Chemical Society, and Mr. K. W. Luckhurst, Secretary of the Royal Society of Arts.

From the Board of Trade we are again glad to see Mr. C. G. Hulse, and from across the border Mr. D. W. B. Scott of the Bradford Textile Society and Mr. W. Hiles of the Engineering Society of that same city. Twin keystones of the industries we, as

a Society, serve are with us in the personages of Mr. G. F. Williams of the Association of British Chemical Manufacturers and Mr. Harry Golden of the Dyers and Finishers' Association.

Mr. R. D. Chorley, our printer, and Mr. L. E. Jones, our Honorary Patent Agent, we regard as members of the family. And now that I am nearer home I must beg to coin collective nouns: there is a veritable pride of past presidents—Dr. C. M. Whittaker, Mr. Fred Scholefield, Mr. Harold Bowen are for this occasion the guests of the Society for which they have done so much; in company with them, and to some extent sharing the same identities, there is a weighty emolument of managing directors.

You will, I am sure, have noticed how amongst our guests almost everybody has attained some distinction within recent weeks of our Annual Dinner and how often we have been able to congratulate them personally. Now this is also true of a number of guests of whom I have made no mention by name so far. I am thinking particularly of those who work in the Society and to whom any welcome from myself would be something of an impertinence. But included in this toast must be those who have received the Gold and Silver Medals of our Society.

In this Society we are not given to conscious pot hunting or sitting around congratulating each other: we get on with the job. And what a magnificent job Dr. P. W. Cunliffe and his colleagues have done in producing the third edition of the Society's fastness tests, which is a handbook of sound practices for the whole world.

Dr. B. V. BOWDEN (*Principal of the Manchester College of Technology*) said that he was proud to reply on behalf of so distinguished a body of guests. Looking around him, the prosperity of the Society was obvious, though it was a pity that specialists in dyeing and finishing should be so drably dressed.

Dyeing and finishing were probably the most scientific part of the textile industry, and he would like to stress the importance of sound technological training. The growth and present expansion of the College of Technology were due to the enterprise and imagination of Manchester Corporation. The College had had a long and honourable connection with the dyeing and finishing trade, and tribute had been paid by Ivan Levinstein to the soundness of the chemical and technical training given to students, in his presidential address to the Society of Chemical Industry in 1903. The new dyehouse is even more magnificently equipped than the old one, but unfortunately it is not attracting a sufficiently large number of British students. The total number of British full-time degree students is about 18 as compared with 14 foreign students; but (and this is much more serious) the number of British research students is falling fast—the total since about 1953 has been less than the number of foreign students; in fact, at the moment it is less than half as many. We have had many meetings with the trade to discuss this lamentable situation, and the trade is becoming aware of the serious implications of this position and is doing what it can to help us. Several large firms have endowed

scholarships, and their help is very much appreciated. In addition, other firms have generously given help to enable their own employees to take courses at the College and read for University degrees.

Since this is the year of the Perkin Centenary, it is obviously most appropriate that we should make a drive to improve the way in which our unrivalled resources are exploited. One of the most significant differences between the educational systems in this country and those on the Continent is that the Continental schools are much more closely integrated than ours with the research institutions on the one hand and with industry on the other. Members of the staff of the research institutions are often part-time Professors Extraordinary in the technological institutions, and so also are prominent industrialists. Moreover, institutions undertake research work for industry on a large scale. I believe that we should introduce this system here, and we can most conveniently begin by making use of the facilities of the dyehouse to allow industrialists to try out experimental processes. We ourselves are prepared to do experimental work for them to the best of our ability.

#### PRESENTATION OF DIPLOMA OF HONORARY MEMBERSHIP TO SIR ROBERT ROBINSON

Mr. CLIFFORD PAINE (*President of the Society*) presented a Diploma of Honorary Membership of

the Society to Sir Robert Robinson, awarded "in recognition of his invaluable services to the Society and his outstanding researches on colouring matters".

#### PRESENTATION OF SOCIETY'S MEDALS

The President then presented the following medals—

<i>Perkin Medal</i>	Mr. J. R. Whinfield
<i>Bar to Gold Medal</i>	Dr. P. W. Cunliffe
<i>Gold Medal</i>	Mr. G. S. J. White
<i>Silver Medals</i>	Dr. H. W. Ellis
	Mr. J. G. Grundy
	Mr. K. McLaren
	Dr. M. E. Probert
	Mr. J. V. Summersgill
	Mr. E. Wilson

#### OTHER AWARDS

The Perkin Medal had been awarded posthumously to Dr. W. H. Carothers for the discovery of nylon.

The Research Medal of the Worshipful Company of Dyers for 1954-1955 had been awarded to Dr. C. H. Giles.

Tokens of acknowledgment had been awarded by the Council of the Society to all members of the Fastness Tests Co-ordinating Committee and the Fastness Subcommittees.

### PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE—XVIII

## Influence of Temperature and Humidity on Fading

P. W. CUNLIFFE

Results are presented of experiments carried out in 1931 on the influence of temperature and humidity on the degree of fading of eighteen dyes on wool cloth.

In the 1934 Fastness Report<sup>1</sup> it was stated that exposures had been carried out at two temperatures, viz. 25°C. and 60°C., and each of these at two different values of relative humidity, viz. 32% and 100%. The details of these experiments, which were carried out in 1931, were not published at the time for various reasons. In view of the present interest in the subject, however, it is felt that it would be useful if the information were now made available.

As stated in the Report<sup>1</sup>, the apparatus consisted of double-walled glass vessels, through the annular space of which water at the required temperature was circulated. Four such vessels were used, each pattern being exposed to a carbon arc under four different conditions simultaneously, viz. (i) 25°C. and 32% R.H., (ii) 25°C. and 100% R.H., (iii) 60°C. and 32% R.H., and (iv) 60°C. and 100% R.H. The patterns were sewn round glass rods suspended from a stopper in the vessel opening, as shown in Fig. 1. The two values of R.H., viz. 32% and 100%, were obtained by means of (a) a saturated solution of magnesium chloride and (b) water respectively, these being

placed in the bottom of the appropriate vessels. As the light from the arc passed through a layer of water before falling on the patterns and as the total volume of air in each vessel was small, it was probable that the temperature and the humidity of the atmosphere surrounding the patterns were satisfactorily controlled at the stated values.

Each pattern was exposed for two periods of time, these being longer for the drier conditions than for the moist conditions. The colours of the exposed and of the unexposed dyeings were then measured in the Guild trichromatic colorimeter<sup>2</sup>, and by means of a formula<sup>3</sup> the time required to produce the same degree of fading under each of the four conditions was calculated. As there were slight differences between the dyes in the degree of fading chosen for the calculation, the figures given are not necessarily a measure of the relative fastness properties of the dyes.

The results obtained on four violet, six red, and eight blue dyes on worsted serge are given in Table I. The table gives also the temperature ratio for each humidity and the humidity ratio for each temperature, as calculated from the corresponding

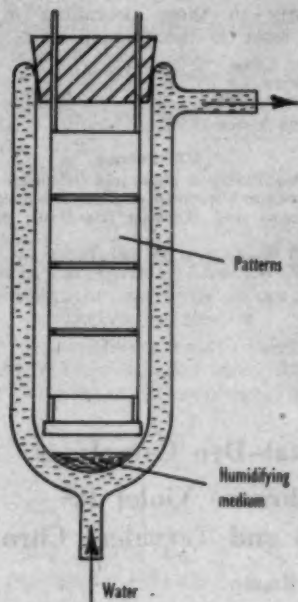


FIG. 1

exposure hours. The range of R.H. ratios is from approx. 1.0 at 25°C. to 4.4 at 60°C., and the range of temperature ratios is from approx. 1.0 at 32% R.H. to 4.5 at 100% R.H.

It will be observed that the temperature ratios are greater than the R.H. ratios with some dyes, while the converse is true with other dyes. Although this comparison is an arbitrary one, it

may serve as a means of classifying the relative behaviour of the dyes examined (Table II).

TABLE II

## Classification of Dyes

## (a) TEMP. EFFECT &gt; R.H. EFFECT

Benzyl Bordeaux B  
Polar Blue G conc.  
Solway Blue RS  
Alizarine Direct Blue A2G  
Brilliant Indocyanine 6B

(b) TEMP. EFFECT  $\approx$  R.H. EFFECT

Lissamine Violet 6BNS  
Acid Magenta  
Acid Scarlet RS

## (c) TEMP. EFFECT &lt; R.H. EFFECT

Lanasol Violet B  
Victoria Violet L  
Azo Geranine 2GS  
Acid Scarlet 3BDS  
Chlorazol Fast Red FGS  
Cloth Fast Red R  
Solway Blue B  
Benzyl Fast Blue L  
Benzyl Fast Blue BL  
Lissamine Blue BFS

As stated in the Report<sup>1</sup>, the eight light standards were chosen for their relatively low sensitivity to moisture during fading. Solway Blue R when tested originally was found to fall in this class and was accepted as Standard 5, a position which it has maintained throughout the

TABLE I

## Fading Times of Wool Dyeings

Dyeing*	Temp.-R.H. (°C.-%)	Hours to Fade				Temp. Ratio		R.H. Ratio		
		60°-100%	60°-32%	25°-100%	25°-32%	100% 32% R.H.	60°C. 25°C.			
VIOLETS										
0.5%	Lissamine Violet 6BNS ...	...	47	73	76	93	1.61	1.27	1.55	1.22
0.5%	Acid Magenta ...	...	23	30	32	30	1.39	1.00	1.30	0.94
0.5%	Lanasol Violet B ...	...	60	140	125	205	2.08	1.46	2.34	1.64
0.5%	Victoria Violet L...	...	40	148	130	370	3.25	2.50	3.70	2.84
REDS										
0.5%	Acid Scarlet RS ...	...	175	334	335	360	1.91	1.08	1.91	1.07
0.5%	Azo Geranine 2GS ...	...	120	260	140	310	1.17	1.19	2.17	2.22
0.5%	Acid Scarlet 3BDS ...	...	130	570	375	600	2.88	1.05	4.38	1.60
0.5%	Chlorazol Fast Red FGS ...	...	310	850	690	860	2.22	1.01	2.74	1.25
1.0%	Cloth Fast Red R ...	...	248	338	255	350	1.03	1.03	1.36	1.37
0.5%	Benzyl Bordeaux B ...	...	143	208	270	315	1.89	1.51	1.46	1.17
BLUES										
—	Polar Blue G conc. ...	...	196	313	398	412	2.03	1.32	1.60	1.03
0.5%	Solway Blue RS ...	...	110	375	500	640	4.54	1.71	3.41	1.28
—	Solway Blue B ...	...	118	370	325	340	2.76	0.92	3.14	1.05
—	Alizarine Direct Blue A2G ...	...	115	270	360	500	3.14	1.85	2.35	1.39
0.5%	Benzyl Fast Blue L ...	...	110	265	217	290	1.97	1.09	2.41	1.33
0.5%	Benzyl Fast Blue BL ...	...	53	172	118	230	2.23	1.33	3.24	1.95
0.5%	Brilliant Indocyanine 6B ...	...	55	155	222	350	4.05	2.25	2.82	1.58
0.7%	Lissamine Blue BFS ...	...	72	165	116	400	1.61	2.43	2.29	3.45

\* The names of the dyes are those in use in 1934

ensuing years. On the other hand, the data now presented indicate that the fading of Solway Blue R is considerably accelerated by moisture. The explanation of the apparent disagreement is to be sought in the difference between the experimental conditions employed in the two investigations. In the original tests the patterns were exposed in boxes containing humidifying solutions, but in the absence of a water screen the temperature of the patterns would rise considerably, resulting in a lowering of the moisture absorbed. These conditions, however, were satisfactory for use in a sorting test and correspond

more nearly to those prevailing in daylight exposures than do the present ones.

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MANCHESTER

(Received 13th March 1956)

#### References

- <sup>1</sup> *Report of the Society of Dyers and Colourists on the work of its Fastness Committee in fixing standards for Light, Perspiration and Washing* (Bradford: the Society, 1934).
- <sup>2</sup> Cunliffe, P. W., *J.S.D.C.*, **45**, 305 (1929).
- <sup>3</sup> Cunliffe, P. W., and Lambert, P. M., *J.S.D.C.*, **47**, 225 (1931).

### COMMUNICATION

## Spectrophotometric Studies on Metal-Dye Complexes

### I—The Reaction between Solochrome Violet RS (2-Hydroxy-5-sulphophenylazo- $\beta$ -naphthol) and Tervalent Chromium

R. B. BENTLEY and J. P. ELDER

An investigation has been made by spectrophotometric and chromatographic methods of the reaction between an *oo'*-dihydroxyazo dye and trivalent chromium salts. The two complexes formed have been separated chromatographically and their spectra measured. Equilibrium constants for the interconversion of the two complexes have been determined, together with the heat, free energy, and entropy changes for this reaction.

#### INTRODUCTION

Metal-dye complexes have been of considerable technical importance for a long time and have consequently been extensively investigated. Three main aspects have been studied, viz. preparation, constitution, and application<sup>1-3</sup>. The present paper represents the first phase of an investigation of the absorption spectra of water-soluble metal-dye complexes in solution and the application of spectrophotometry to obtain their stability constants and thermodynamic data.

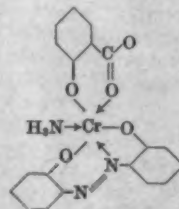
Ions or molecules forming metal complexes are usually referred to as *ligands* and are classified according to the number of points of attachment to the central metal atom. A simple molecule, such as ammonia, with one co-ordinating group (monodentate ligand), may attach itself to only one co-ordination position of the metal. Molecules such as ethylenediamine, with two co-ordinating groups, i.e. bidentate ligands, may occupy two co-ordination positions, and so on.

Schwarzenbach and his co-workers<sup>4</sup> examined complexes of *oo'*-dihydroxyazo dyes similar to the one used in the present work. Chelation with alkaline-earth metals was studied in connection with the use of these dyes as metal indicators, responsive to metal ion concentration in a manner analogous to the response of acid-base indicators to hydrogen-ion concentration. The complexes formed with these bivalent metals are only of the type in which one dye molecule is bound to one metal atom (1:1 complexes).

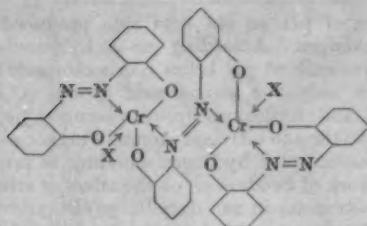
With trivalent chromium as the central atom, *oo'*-dihydroxy-, *o*-hydroxy-*o'*-carboxy-, and

*o*-hydroxy-*o'*-amino-azo dyes are well known to behave as tridentate ligands, one azo nitrogen acting as a donor in addition to the binding due to the salt-forming and co-ordinating *ortho-ortho'* groups<sup>1</sup>. Thus with six positions available around the chromium atom, two types of complex may be formed, a 1:1 complex and a 2:1 complex, the latter containing two molecules of dye bound to the central chromium atom. The particular complex formed depends on the conditions of reaction and on the presence of other donor groups which may enter the inner co-ordination sphere ( $H_2O$ ,  $NH_3$ ,  $Cl$ , etc.).

It is also possible to introduce larger groups along with the dye to form mixed complexes. Thus a bidentate group such as a salicylic acid residue and one dye molecule may form a complex of the type<sup>5</sup>—



Polynuclear complexes of the 3:2 type have also been reported<sup>6</sup>, as water-insoluble precipitates from reaction with disalicylatochromic acid and its ammonium or ammine salts, and assigned structures of the type—



where X represents a monodentate ligand.

#### SPECTROPHOTOMETRIC METHODS OF INVESTIGATING COMPLEX FORMATION

Bjerrum<sup>10</sup> has shown that co-ordination of donor groups (A) to a metal atom (M) may be represented as a series of "step equilibria" of the type—

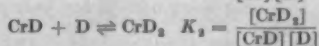
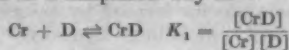


for which equilibrium constants may be defined, e.g.—

$$K_t = \frac{[MA_t]}{[MA_{t-1}][A]}$$

Bjerrum developed potentiometric and optical methods for determining these equilibrium constants, and their significance in the study of complex ions is now widely recognised<sup>11</sup>.

If complexes of the 2:1 and 1:1 types were formed simultaneously from a dye D and chromium there would be two step equilibria, which may be represented in a simplified way as—



$K_1$  and  $K_2$  could then be calculated from a knowledge of the equilibrium concentrations of the unreacted dye and chromium and of the two complexes.

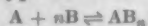
Spectrophotometric examination of the products obtained from a series of reaction mixtures containing varying proportions of metal and chelating agent may be used to decide whether one or more complexes are formed. In favourable cases the proportions of metal and chelating agent present in a given complex may be found. Several methods of treating the data have been reported<sup>12,13</sup>, but the most general is Job's method of "continuous variations" as extended by Vosburgh and Cooper<sup>14</sup>. It can be shown by simple application of the law of mass action that, if only a single complex of formula  $MA_n$  is formed, and the reactants are mixed in varying proportions, whilst the sum of the molar concentrations is kept constant, then the complex reaches its maximum equilibrium concentration at the composition represented by the formula. This initial proportion of reactants may be found by measuring some property of the equilibrium mixture such that the molar contributions of complex and reactants differ, the individual contributions being linearly

AO

related to concentration. The most widely used physical property, which in general fulfils these conditions, is the absorption of monochromatic light, ultraviolet radiation, etc. A large number of investigations of stoichiometry by this method have been reported, including copper lakes of azo dyes\*. If Beer's law is obeyed, the concentration of the complex is usually taken to be proportional to the difference between the optical density of the final equilibrium solution and the optical density calculated on the assumption of no reaction. This difference, usually called the "Y function", plotted against molar composition of the initial reaction mixture, would reveal the maximum, for a complex having a large stability constant, as the intersection of two straight lines. Here the unreacted part is essentially the stoichiometric excess of one or other reactant. For less stable complexes, i.e. in the more general case, the plot forms a curve in the region of the stoichiometric composition, and extrapolation of the linear portions is necessary. For a dye forming only a 1:1 complex with chromium, the Y function would be expected to reach a maximum at a mole fraction of dye  $x = 0.5$ . If only a 2:1 complex were formed, the maximum would be expected at  $x = 0.67$ .

For simultaneous formation of more than one complex, Vosburgh and Cooper have shown that the method can be applied successfully only assuming either (a) that the concentration of one of the complexes is negligibly small when the reaction mixture reaches the stoichiometric composition of the other complex; or (b) that wavelengths can be chosen at which the absorption of one complex is negligible compared with that of the other. In most work on metal-ion complexes of highly coloured ligands, the absorption of the metal ion (or its ion-pair complexes\*) may be neglected in comparison with the absorption of the other reactant and its metal complexes over a wide wavelength range.

One further point may be noted here. In all equilibria of the type—



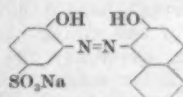
in which A and  $AB_n$  absorb strongly compared with B, there is generally a shift of the absorption band to longer or shorter wavelengths on formation of  $AB_n$ , so that the extinction-coefficient curves of A and  $AB_n$  will overlap at one or more wavelengths. At the points of equal extinction coefficient, mixtures of A and B containing a constant amount of A will always have the same absorption, independent of the amounts of B and of the compound  $AB_n$  at equilibrium (or at any stage of the reaction). Such crossing points are well known in the case of two-colour acid-base indicators, for which a series of absorption curves all passing through well defined crossing points—*isobestic* or *isosbestic* points—are obtained at varying pH<sup>15</sup>. Where more than one equilibrium is established, well defined isobestic points will not generally be obtained. However, in the case of chromium-dye complex formation the molar extinction

\* Ion-pair complexes are discussed more fully on p. 334.

coefficients of D and CrD at their isobestic points were not very different from half the molar extinction coefficient of CrD<sub>2</sub> at these wavelengths, the deviations of reaction mixtures at the crossing points would not be very large.

#### INVESTIGATION OF COMPLEX FORMATION BETWEEN AZO DYES AND TRIVALENT CHROMIUM

Preliminary work was carried out on Solochrome Red B (C.I. 216) and has been presented elsewhere in summary form<sup>16</sup>. The present paper is mainly concerned with the results for Solochrome Violet RS (C.I. 169), 2-hydroxy-5-sulphophenylazo- $\beta$ -naphthol—



From the work of Drew and Fairburn<sup>1</sup>, this dye is known to form 1:1 and 2:1 complexes. Fig. 1 shows the absorption spectrum in water at three concentrations, from which it appears that Beer's law is obeyed fairly well over the whole wavelength range. Molar extinction coefficients at 500 m $\mu$ . (near the peak wavelength in the visible spectrum) show that Beer's law holds well within experimental error over a wider concentration range (1.0–25.0  $\times 10^{-4}$ M.).

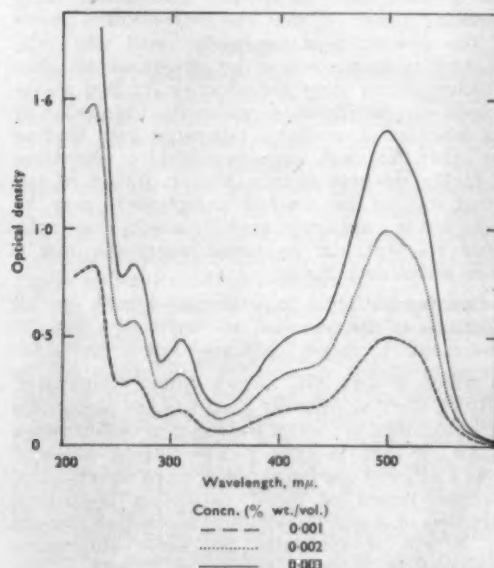
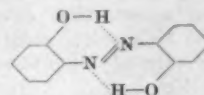


FIG. 1.—Absorption Spectra of Solochrome Violet RS (distilled water as solvent; cell length 1.0 cm.)

Extinction coefficients of dyes of this type change considerably when their solutions are heated, indicating dissociation of molecular aggregates. However, the process is rapidly reversible, and since all solutions were examined at room temperature, no errors due to this effect were encountered. Further, addition of an electrolyte such as potassium nitrate, up to an ionic strength of 0.5, had little effect on the absorption spectrum. Similarly,

variation of pH on the acid side produced only small changes. According to Schwarzenbach<sup>2</sup>, these dyes exist at pH below 6 in mesomeric forms in which only the strong acid group SO<sub>3</sub>Na is ionised, the hydroxyl groups being hydrogen-bonded to the azo nitrogen atoms. Further support for intramolecular hydrogen bonding is provided by the work of Brode *et al.* on the effect of structure on phototropism in azo dyes<sup>17</sup>. *oo'*-Dihydroxyazo dyes were shown to be non-phototropic, and it was supposed that *trans*  $\rightarrow$  *cis* changes were prevented by the fixation of the molecule in the *trans* form by hydrogen bonding—



(see also Conmar Robinson<sup>18</sup>).

It is interesting that where *trans*  $\longleftrightarrow$  *cis* phototropism occurs, both about N:N bonds in azo dyes and about C:C bonds in thioindigo dyes, the *cis* form has a peak at shorter wavelengths and of lower extinction coefficient. Similar effects are found with *trans*  $\longleftrightarrow$  *cis* conversion in polyenes<sup>19</sup>, and are attributed to the smaller extension of the polarisable conjugated system in the *cis* form. This point will be discussed later in connection with the absorption spectra of the complexes.

In investigations of the reactions of metal ions, it is now realised that account must be taken of all the species which may be present in solution. Transition metals, in particular, exist as simple hydrated ions only under special conditions, and are generally present also in the form of various complex ionic species formed by association with anions present, including hydroxyl<sup>20</sup>. Species formed in this way were named *ion-pair complexes* by N. Bjerrum.

Solutions of chromic perchlorate and nitrate at low pH contain mainly the simple chromic ion, associated with six tightly bound water molecules, for perchlorate and nitrate anions show the least tendency to complex with metal ions. Concentrated solutions of these salts are violet. Their colour is associated with transitions of electrons among the *d* levels of the chromium atom, which lead to the appearance of two low-intensity absorption bands in the visible spectrum<sup>21</sup>. Such intra-valency-shell transitions are not strongly affected by external influences, such as ion-pair complex formation.

Chromic nitrate and perchlorate have also a more intense absorption band in the ultraviolet, which corresponds to transfer of an electron to the metal ion from one of the water molecules in its hydration sphere. This electron-transfer band is displaced towards the visible by anion association, e.g. with SO<sub>4</sub><sup>2-</sup>, Cl<sup>-</sup>, CNS<sup>-</sup>, OH<sup>-</sup>, and the electron transfer then involves the anion complexed.

Hydroxyl complexes are formed on diluting chromic nitrate or perchlorate solutions, the species formed being Cr(OH)<sup>2+</sup>, Cr(OH)<sub>2</sub><sup>+</sup> (both hydrated). It is unfortunate that the molar extinction coefficients of the dye are so large compared with those

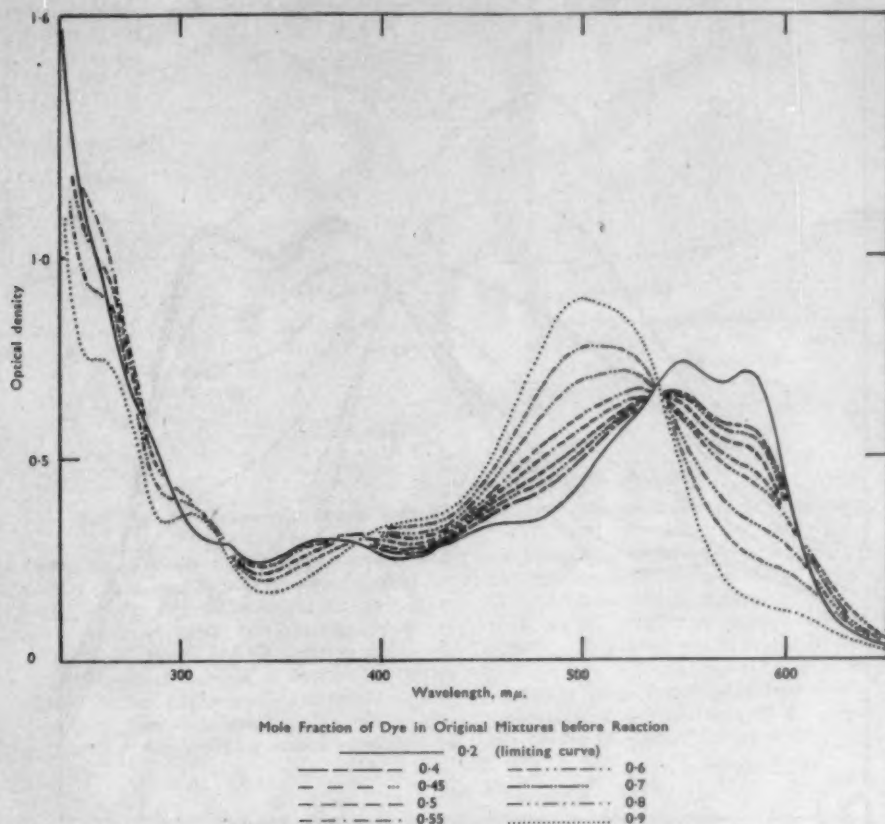


FIG. 2—Absorption Spectra of Equilibrium Mixtures of Solochrome Violet RS and Potash Chrome Alum in Distilled Water at 100°C. (all mixtures diluted to  $0.5 \times 10^{-4}$  M. based on original dye concentration; cell length 1 cm.)

of the chromium salts in the region of the electron-transfer bands that it is not possible to study the displacement of the latter in these reactions.

In view of the fact that complex formation with dye may entail competition with other complexes present in solutions of chromic salts, a comparison was made of reactions with chromic nitrate and with chrome alum. The effects of additions of sodium perchlorate, potassium nitrate, and potassium sulphate were also studied.

Fig. 2 and 3 show the spectra obtained in the application of the method of continuous variations to reaction mixtures of Solochrome Violet RS with chrome alum and with chromic nitrate respectively. To demonstrate isobestic points, all solutions were diluted, after equilibrium had been reached, to  $0.5 \times 10^{-4}$  M., based on their original dye content. At first sight the curves appeared to have fairly satisfactory isobestic points, especially in the visible region. On closer examination, however, it became apparent that more than one complex is formed unless the ratio of chromium to dye is at least 4:1.

The main evidence for this is as follows—

(a) For low Cr:D ratios there is a crossing point at about 540 mμ., but for high ratios it varies from mixture to mixture. More pronounced

variations occur in the long-wave region above 600 mμ.

(b) With a 4:1 ratio ( $x = 0.2$ ) in both reactions, a limiting curve is attained with a characteristic double peak, presumably the absorption curve of the 1:1 complex. However, in the chromic nitrate reaction the curves for  $x = 0.45$  and 0.40 are slightly higher around the region of this double peak.

(c) The continuous-variations plots (Fig. 4) show considerable curvature above  $x = 0.5$ , and extrapolation is not satisfactory.

(d) If only one complex were formed, the limiting double-peak curve representing its absorption spectrum, conventional two-component analysis should be possible at a suitable wavelength, e.g. 500 mμ. or 580 mμ., to determine the amounts of complex and unreacted dye in the various equilibrium mixtures. Consistent results could not be obtained in this way.

At this stage it was obvious that the spectrophotometric work must be supplemented by separation of the complexes from each other and from the dye. This was achieved by a quantitative chromatographic method using cellulose powder, details being given in the experimental section. By chromatographing a known volume of an

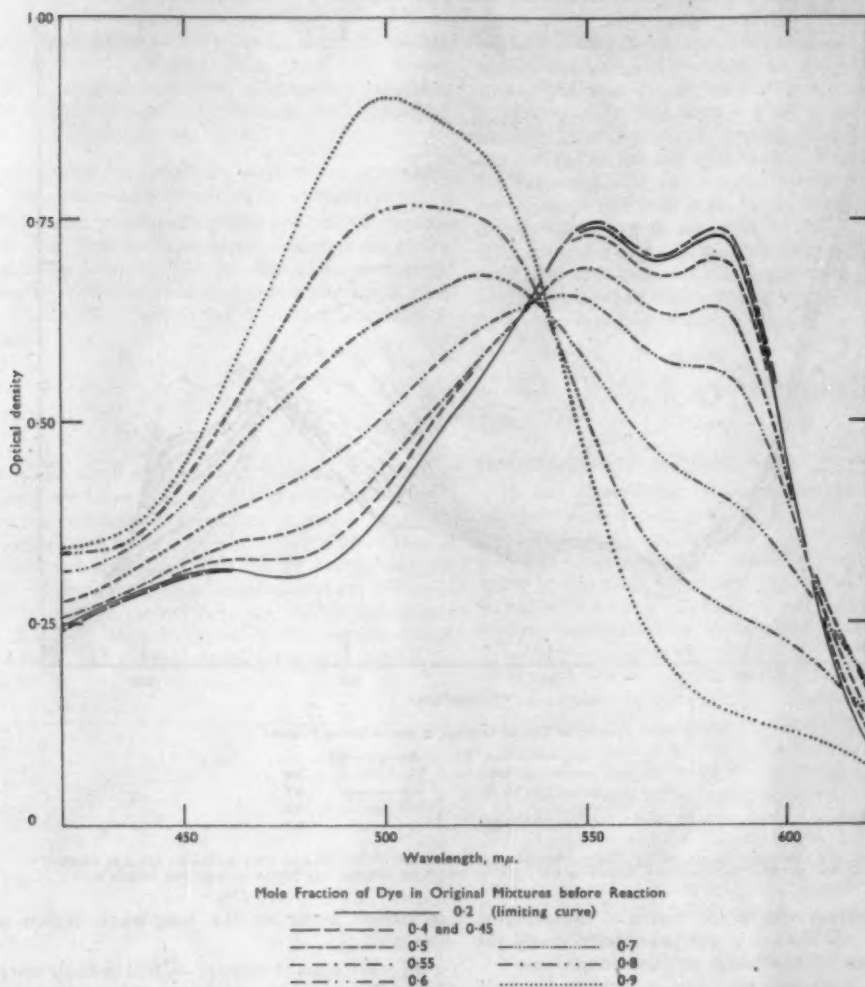


FIG. 3—Absorption Spectra of Equilibrium Mixtures of Solochrome Violet RS and Chromic Nitrate in Distilled Water at 100°C. (all mixtures diluted to  $0.5 \times 10^{-4}$  M. based on original dye concentration; cell length 1 cm.)

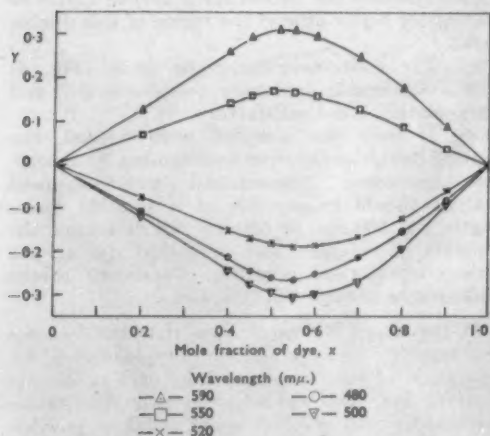


FIG. 4—Continuous-variations Plots for the Reaction of Solochrome Violet RS with Chromic Nitrate

equilibrium mixture, three solutions were obtained containing the free dye, 1:1 complex, and 2:1 complex respectively, and their absorption spectra were measured. The volumes of these solutions being known, it was then possible to calculate the contributions of the above three species to the optical density of the original equilibrium mixture or to its optical density after dilution to  $0.5 \times 10^{-4}$  M. based on the original dye content before reaction.

Fig. 5 and 6 are typical of these analyses, relating to a mixture refluxed to equilibrium at 100°C. and 80°C. respectively. The sums of the contributions are drawn for comparison with the mixture curves, and the agreement is satisfactory. Duplicate analyses agreed within experimental error. From these results concentrations of dye and the two complexes were calculated using molar extinction coefficients obtained as follows—

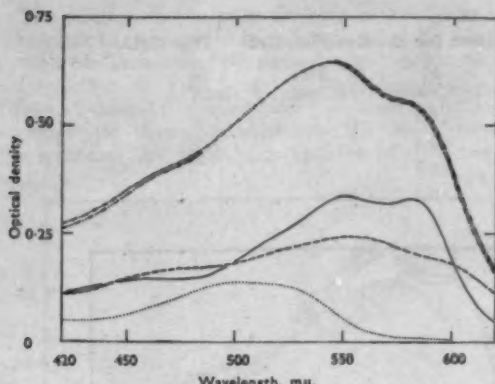


FIG. 5—100°C.

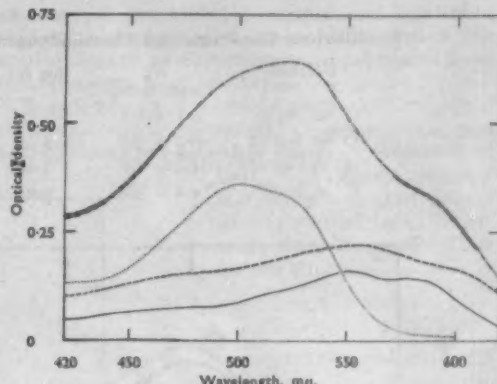


FIG. 6—80°C.

..... Unchanged dye (D)  
 — CrD  
 - - - CrD<sub>2</sub>  
 . . . Mixture  
 - - - - Sum of contributions of D + CrD + CrD<sub>2</sub>

FIG. 5 and 6—Analysis of Solochrome Violet RS-Chromic Nitrate Equilibrium (Mixture "D"—Table III)

**1:1 COMPLEX**—A chromatograph fraction containing only this complex was decomposed by boiling with oxalic acid. The molar concentration was then calculated from the absorption of the regenerated dye. From the absorption spectrum of the undecomposed fraction, it was then possible to calculate molar extinction coefficients. They were close to values calculated on the alternative assumption that the limiting curve obtained at

azo nitrogen content being no absolute indication of material capable of combining with chromium. The calculated totals of free and combined dye are close to  $0.5 \times 10^{-4}$  M. except for mixture "D" at 80°C. This latter result might be due to destruction of dye by prolonged boiling, as there is a relatively large fraction of free dye. In view of these results, calculations of  $K_1$  are not possible except for mixture "D" at 80°C.

TABLE I  
 Analytical Results for Diluted Equilibrium Mixtures

(All concn. in M.  $\times 10^4$ )

Mixture	Dye	CrD <sub>2</sub>	CrD	Total Dye	Combined Cr (calc.)	Total Cr added
"C" at 100°C.	0.151	0.113	0.105	0.482	0.218	0.208
"D" at 100°C.	0.063	0.098	0.236	0.485	0.334	0.323
"D" at 80°C.	0.167	0.090	0.104	0.451	0.194	0.323
"E" at 100°C.	0.033	0.057	0.360	0.507	0.417	0.397

high Cr:D ratios represents the absorption spectrum of  $0.5 \times 10^{-4}$  M. 1:1 complex (see Fig. 3).

**2:1 COMPLEX**—A chromatograph fraction was decomposed as for the 1:1 complex, and the molar concentration and the extinction coefficients were calculated in a similar way. It was further shown by decomposing equilibrium mixture "D" ( $x = 0.60$  mole fraction of dye before reaction) with oxalic acid that the original dye is regenerated with a loss of only about 2%.

Table I shows the final analytical results for the diluted reaction mixtures. Except for the 80°C. reaction mixture, the amount of unreacted chromium is obviously very small; in fact, owing to accumulation of errors, the combined chromium appears to be slightly greater than the chromium added for all three mixtures at 100°C. The technique had been refined to reduce manipulative and instrumental errors to the minimum. The discrepancies may therefore be due to the fact that the purity of the dye is difficult to assess, even

Table II gives values of  $K_2$  and derived thermodynamic quantities for the reaction—



To calculate the heat of reaction, only data on mixture "D" at 80°C. and 100°C. were used. This value depends merely on analytical accuracy, being independent of the values assumed for the molar extinction coefficients, which cancel out, leaving—

$$\begin{aligned}
 -\Delta H &= \frac{2.303 R}{(x_1' - x_1)} \log \frac{K_2}{K_2'} \\
 &= \frac{2.303 R}{(x_1' - x_1)} \log \frac{d_{\text{CrD}_2} \times d'_{\text{CrD}} \times d'_D}{d'_{\text{CrD}_2} \times d_{\text{CrD}} \times d_D}
 \end{aligned}$$

where the  $d$ s are the optical density contributions of the complexes and the dye to the optical density of the diluted 100°C. equilibrium mixture and  $K_2$  is the equilibrium constant at 100°C.; the primed symbols are corresponding values for the 80°C. equilibrium mixture.

TABLE II  
Equilibrium Constants and Thermodynamic Data for the Reaction  $\text{CrD} + \text{D} \rightleftharpoons \text{CrD}_2$

Mixture	$K_2$ (litres/mole)	$\log K_2$	$\Delta G$ (kcal./mole)	$\Delta H$ (kcal./mole)	$\Delta S$ (cal./°c.)
"C" at 100°C.	$5.1 \times 10^3$	3.71	-6.3	—	—
"D" at 100°C.	$5.5 \times 10^3$	3.74	-6.4	+3	+25
"D" at 80°C.	$4.3 \times 10^3$	3.64	-5.9	—	—
"E" at 100°C.	$4.4 \times 10^3$	3.64	-6.3	—	—

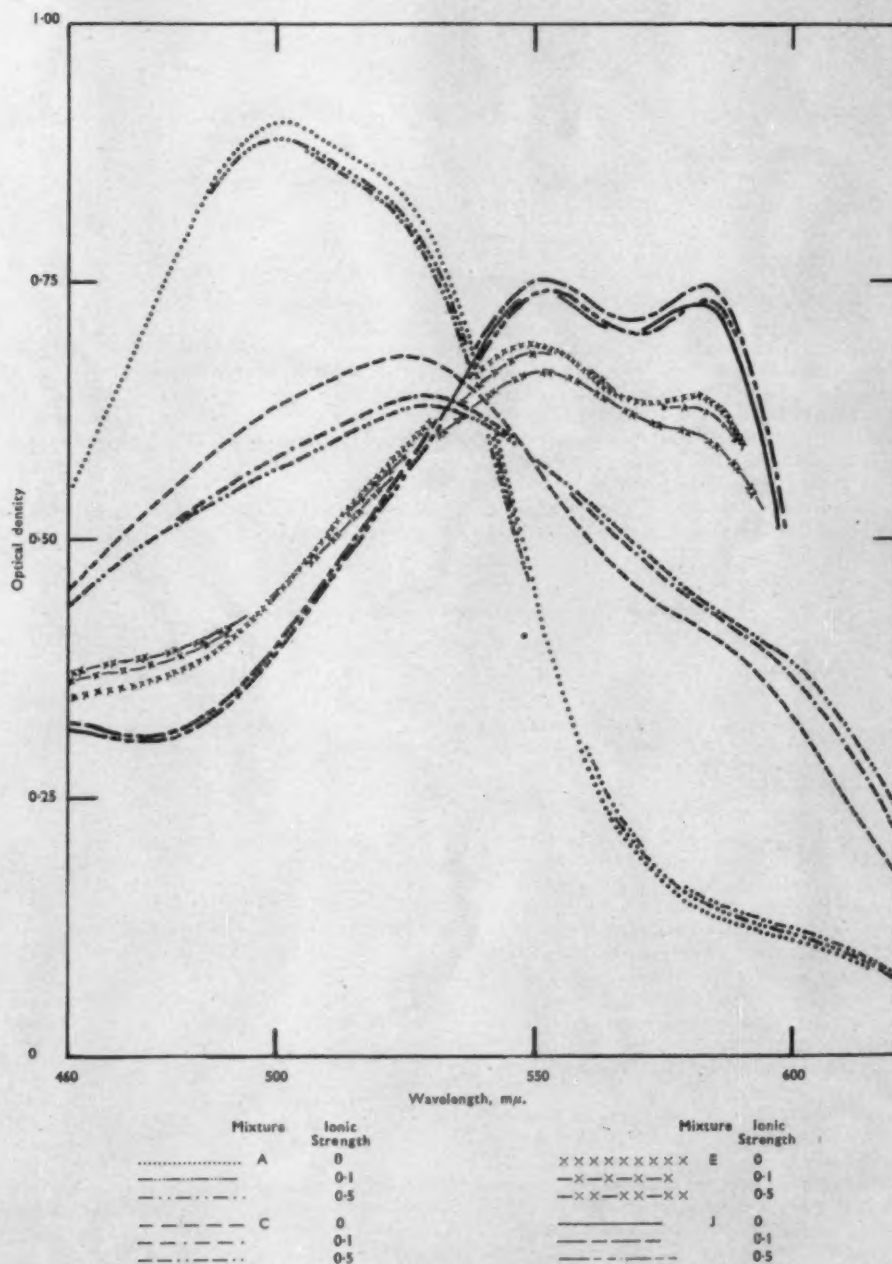


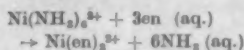
FIG. 7.—Effect of Added Electrolyte (Potassium Nitrate) on the Solochrome Violet RS—Chromic Nitrate Equilibrium at 100°C.

The dilution factor (mole fraction of dye  $\times 20$ ), which has to be included in calculations of  $K_2$ , also cancels out here, being the same for both mixtures.

Inspection of Fig. 5 and 6 and the data in Tables I and II shows that the most reliable analyses are those for mixtures "C" and "D". For mixture "E" (0.55 mole fraction of dye) the

amounts of dye and 2:1 complex are small and subject to experimental errors, even though the optical density contributions were calculated from measurements in 4-cm. cells.

Heat is absorbed in the formation of  $\text{CrD}_2$  from  $\text{CrD}$ , and the entropy effect opposes this and actually leads to a large free energy of formation of  $\text{CrD}_2$ . A large entropy increase is typical of chelate ring formation by displacement of smaller groups. Thus for the reaction—



(en = ethylenediamine)  $\Delta S = 24 \text{ cal./}^\circ\text{C.}$  From the analysis of mixture "D" at  $80^\circ\text{C.}$   $K_1$  may be calculated as  $4.0 \times 10^3$  litres/mole, and the two constants are thus of the same order of magnitude at this temperature.

#### EFFECT OF ELECTROLYTE AND pH ON THE REACTION

Fig. 7 shows curves for four diluted equilibrium mixtures obtained by carrying out the reaction at the boil in the presence of potassium nitrate at three ionic strengths in each case. Added electrolyte does not produce large changes. The effect seems to be mainly that a slightly greater amount of 2:1 complex is produced, especially for mixture "C", in which the initial proportions of chromium and dye are closest to the stoichiometric ratio for this complex.

Fig. 8 shows corresponding curves obtained with sodium acetate-hydrochloric acid buffers at an ionic strength of 0.2. Allowing for the salt effects of the added electrolyte, lowering the pH leaves more unchanged dye, mainly at the expense of 1:1 complex. Rate studies on the formation of the 1:1 complex of Solochrome Red B have shown that the effect of lowering the pH, especially in the range 4–2, by the addition of perchloric acid, is to retard the reaction. Presumably this is due to diminution in the amounts of the ion-pair complexes,  $\text{Cr}(\text{OH})_2^+$  and  $\text{Cr}(\text{OH})_3^+$ , by conversion to  $\text{Cr}(\text{H}_2\text{O})_6^{3+}$ . It should be noted that the

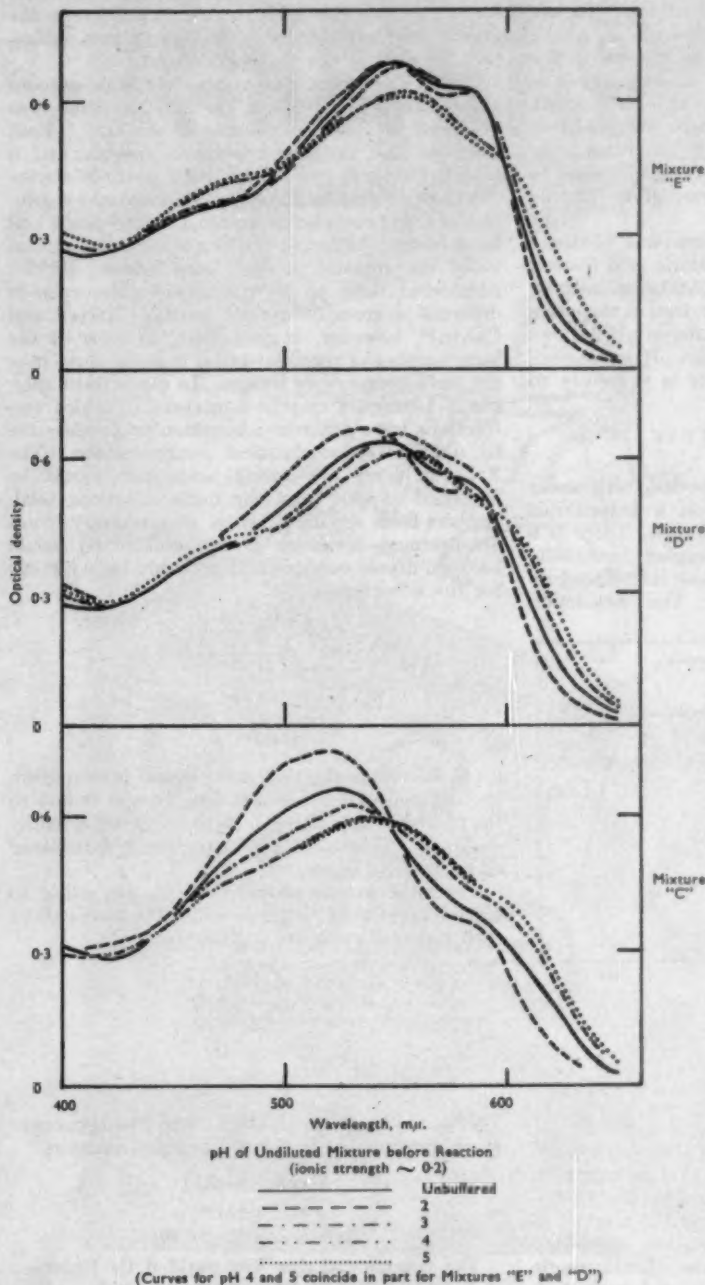


FIG. 8—Effect of Added Electrolyte Buffers on Solochrome Violet RS-Chromic Nitrate Equilibrium at  $100^\circ\text{C.}$  (optical densities (1 cm.) for mixtures diluted to  $0.5 \times 10^{-4}\text{M.}$  based on the original dye concentration)

1:1 complex is not decomposed at any appreciable rate even at pH 1 and 100°C.

The reaction of Solochrome Violet RS with chrome alum proceeds to a much smaller extent than that with chromic nitrate (Fig. 2 and 3). This is presumably due to the presence of stable sulphatochromic ion complexes in the heated chrome alum solution, which is borne out by the effect of addition of potassium sulphate to the reaction mixtures containing chrome alum. As the concentration of sulphate ion is increased, the equilibrium conversion of dye to complexes is reduced. The effect is appreciable at ionic strength 0.1 and much larger at ionic strength 0.5. Addition of sodium perchlorate, however, produces only small changes at ionic strength 0.1, and increase to ionic strength 0.5 produces negligible further change in the curves.

Finally, an equilibrium mixture was obtained at pH 2 by the addition of perchloric acid instead of the buffer to the mixture containing initially 0.55 mole fraction of dye. Comparison of the curve with the curve obtained with a buffer at pH 2 shows a smaller extent of reaction as the pH is lowered, and that the effect of electrolyte is primarily to produce more 2:1 complex.

#### ABSORPTION SPECTRA OF THE DYE AND ITS COMPLEXES

Fig. 9 shows the absorption spectra, with molar extinction coefficients plotted on a logarithmic scale for comparison of curve shapes. There is a flattening of the curves on complex formation, which is the reason for the dullness introduced by chroming dyes on fabrics<sup>22</sup>. The extinction

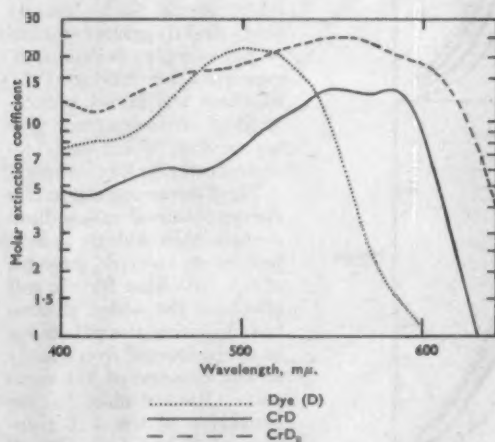
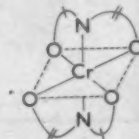


FIG. 9—Spectral Extinction Coefficient Curves for Dye and Complexes

coefficient per mole of dye at the maximum decreases in the order—dye > 1:1 complex > 2:1 complex. Apart from this and the bathochromic shift, there is no change in the number of absorption bands on complex formation, suggesting little fundamental change in the chromophoric system and probably retention of the *trans* configuration about the azo nitrogen double bond. No satisfactory theory of the absorption spectra

of azo dyes is available at present and it is, therefore, difficult to draw definite conclusions. The absorption process associated with the band of longest wavelength in diazo compounds is transfer of an atomic nitrogen electron to an anti-bonding NN  $\pi$  orbital. The energy of this latter orbital is lowered in azo dyes by conjugation with the aromatic rings. Chelation with a metal atom would further reduce the energy difference between the ground and excited states and result in a bathochromic shift of the absorption band.

The 1:1 complex was apparently homogeneous chromatographically, but the 2:1 complex was obtained in two fractions (see p. 342). Both fractions had identical absorption spectra, and it is difficult to reconcile this with possible stereochemical differences. Pfeiffer<sup>23</sup> has claimed resolution of a 2:1 complex of an azo dye into *dextro* and *laevo* forms. Although the *dextro* form had almost twice the rotation of the *laevo* isomer, Pfeiffer considered them to be optical enantiomorphs of different degrees of optical purity. Martell and Calvin<sup>24</sup>, however, suggest that, in view of the large number of possible optical isomers, these may not have been mirror images. In the present case, the 2:1 complex may be a mixture, of which two fractions have different adsorption properties, due to different stereochemical configurations. The 2:1 complexes are strong acids and would be adsorbed as ions from the buffer solutions used. A *trans* form having a centre of symmetry (*trans* arrangement of donor groups and rings) would have no dipole moment and probably little affinity for the adsorbent—



(in this formula the tridentate ligand is simplified, the difference between the two groups linked to the co-ordinating nitrogen atom being indicated by  $\rightarrow$  and  $\leftarrow$ ). This form would require non-coplanar rings in the dye molecule.

The other most probable form, according to Martell and Calvin, is one in which the bonds about each nitrogen atom are coplanar—



This would be polar, and its optical enantiomorphs would presumably be formed in equal amounts.

## Experimental

### MATERIALS

#### Solochrome Violet RS

The commercial dye was purified by Robinson and Mills' method of salting out with sodium acetate<sup>25</sup>. Removal of sodium acetate was found to be difficult. Analytical data (Weiler and Strauss,

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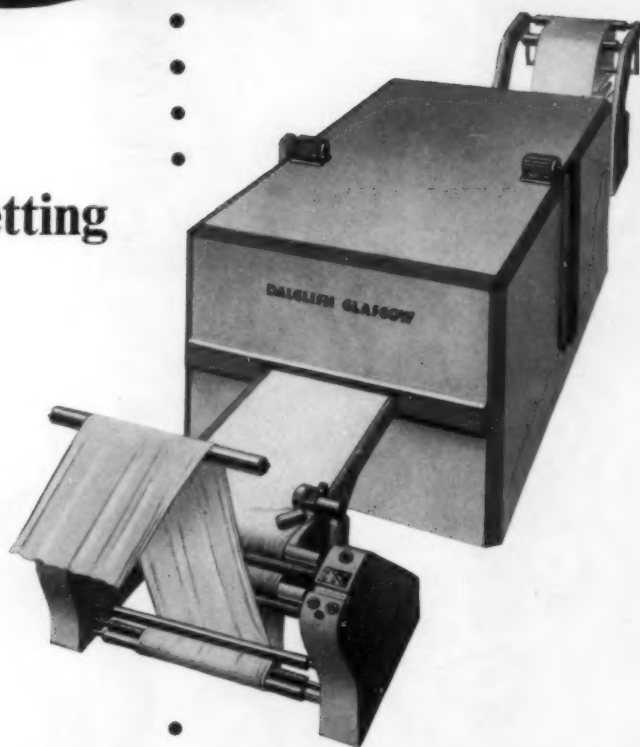


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A. 38

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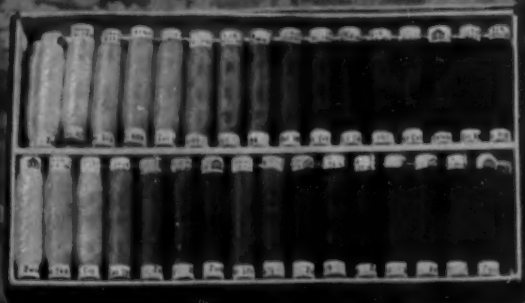
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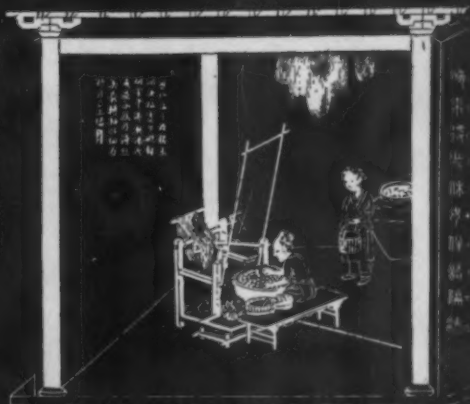
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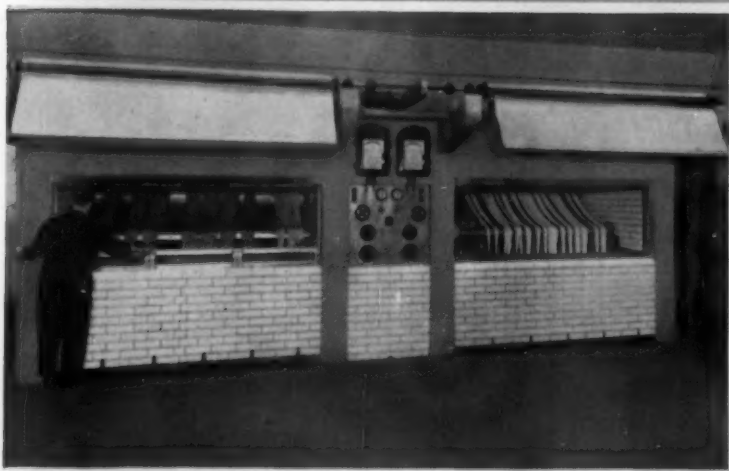
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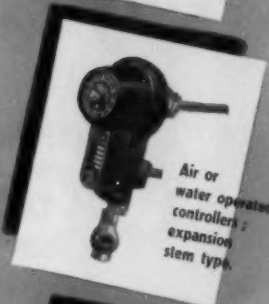
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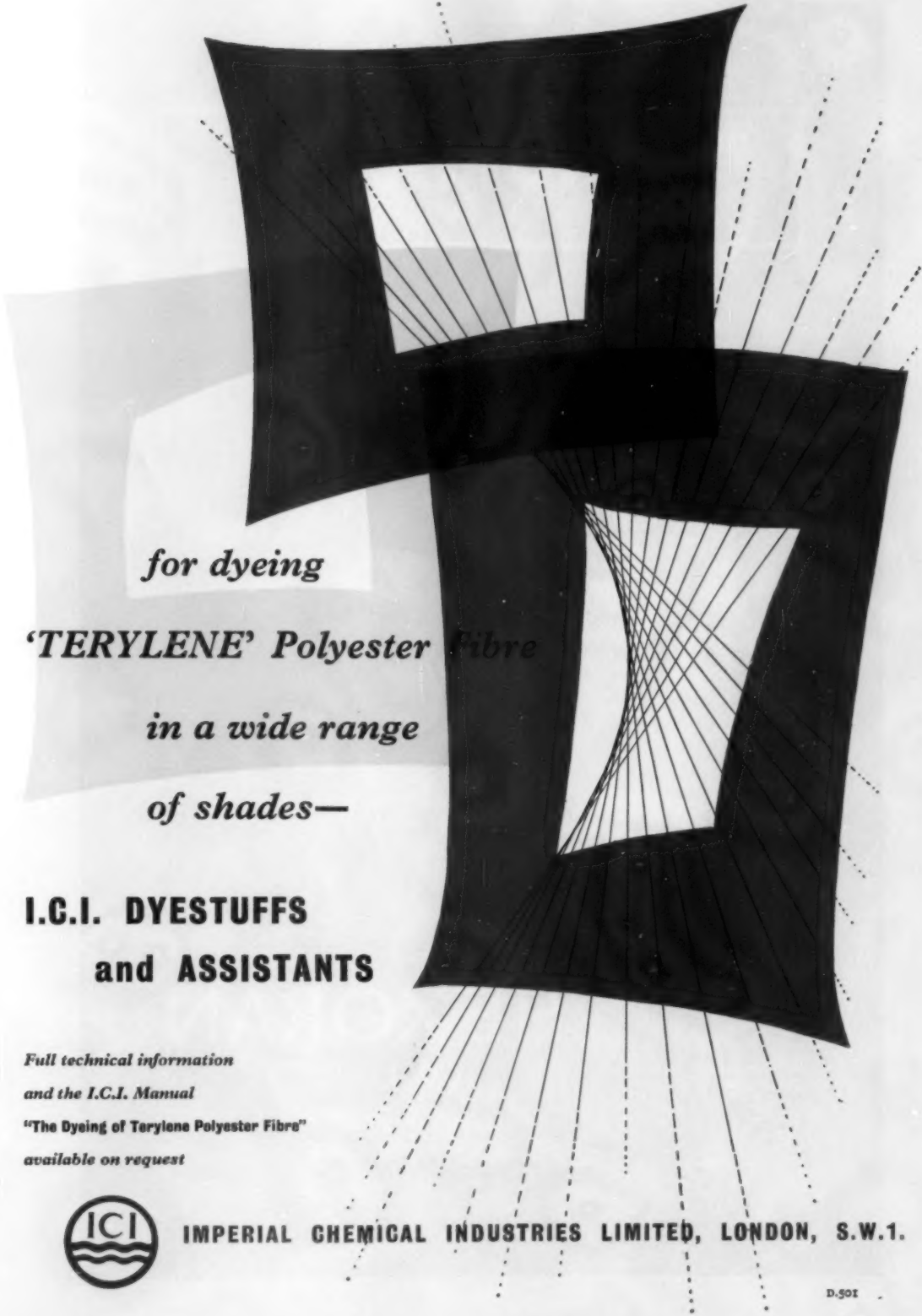


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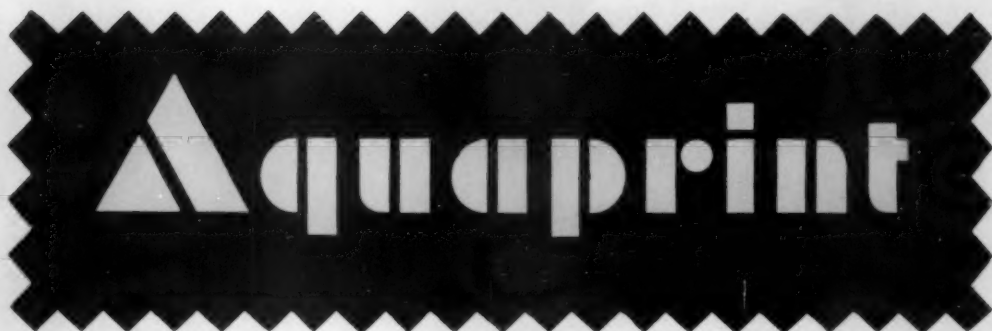
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Oxford) indicate a dye purity of 90% with 10% sodium acetate. 90% purity was also obtained by titanous chloride titration, and this figure is assumed throughout.

#### Chromic Nitrate

The B.D.H. material was used without further purification. Purity as  $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O} = 97.1\%$  based on Cr content.

#### Potash Chrome Alum

The B.D.H. AnalaR grade was used. Purity as  $\text{Cr}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 \cdot 24\text{H}_2\text{O} = 100.7\%$  based on Cr.

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Whatman standard grade, ashless, chemically pure, manufactured by W. & R. Balston Ltd., Maidstone, Kent, specially for chromatographic work. Obtainable from laboratory suppliers.

#### ABSORPTION SPECTRA

All measurements were made with a Uvispek (Hilger) spectrophotometer. Quartz- and glass-prism measurements in the visible spectrum did not differ significantly, as the spectra showed no sharp bands.

#### METHOD OF CONTINUOUS VARIATIONS

Mixtures were made up as in Table III and refluxed to equilibrium (20 hr.). After rapid cooling to room temperature, dilutions were made in two stages to  $0.5 \times 10^{-4}$  M. based on the original dye concentration, and absorption spectra were measured in 1-cm. cells.

Complex formation decreases pH by replacement of hydrogen ions from donor groups. Values of pH for the initial mixtures were all around 4, and the final pH values before and after dilution are included in Table III.

Experiments involving addition of electrolytes or buffers were based on the same scheme, except that the volume of water was reduced and appropriate amounts of concentrated electrolytes or buffers added to give the required ionic strength or pH in the final 100 ml. of reaction mixture.

Dye solution ( $0.5 \times 10^{-3}$  M.) refluxed alone for 20 hr. suffers approx. 10% loss, either by destruction to colourless materials or by adsorption by ion exchange on the walls of the Pyrex glass vessel. This loss was not increased by bubbling oxygen through the solution, nor decreased by bubbling nitrogen through. It is probably due to heterogeneous decomposition on the walls of the flask (see also Harrow and Jones<sup>23</sup>). It has been noted above that the loss is negligible in mixtures containing enough chromium to convert a large part of the dye to one or both complexes.

Vosburgh and Cooper's  $Y$  values<sup>14</sup> are calculated from the difference—

(Optical density of diluted equilibrium mixture

— Optical density of  $0.5 \times 10^{-4}$  M. dye)

at any particular wavelength required. This difference is multiplied by the dilution factor appropriate to the mixture concerned, viz. initial mole fraction of dye  $\times 20$ . Since the factor 20 is constant throughout, it was omitted from the calculations for Fig. 4.

#### CHROMATOGRAPHIC SEPARATION OF DYE AND COMPLEXES

In the quantitative method finally adopted, a four-section jointed Pyrex glass column 3 ft. 6 in. long and of internal diameter 1 in. was packed with cellulose powder by the slurry method with water. Before adsorption the column was buffered to the pH of the equilibrium mixture to be separated. By adsorption from the equilibrium mixtures (diluted to 250 ml.) and elution with buffer at the same pH, a first, sharp-fronted violet band was brought into the lowest (6-in.) section of the column. This was the 2:1 complex. A second, more diffuse, violet band was then seen in one or both of the central 12-in. sections, according to the amount present. This was the 1:1 complex. The dye was adsorbed strongly in the top 12-in. section of the column, which contained only a 4-in. length of adsorbent. The column sections were then disconnected and the fractions separately eluted. The dye was removed by elution with water, and the complexes with buffer. In this way,

TABLE III  
Details of Reaction Mixtures

Mixture	A	B	C	D	E	F	G	H	J
Chromium, g.-atoms $\times 10^{-4}$	0.10	0.20	0.30	0.40	0.45	0.50	0.55	0.60	0.80
Dye, moles $\times 10^{-4}$	0.90	0.80	0.70	0.60	0.55	0.50	0.45	0.40	0.20
Dye soln. (0.1% wt./vol.), ml.	36.6	32.5	28.45	24.4	22.35	20.35	18.3	16.25	8.15
Chromic salt ( $10^{-3}$ M.), ml.	10.0	20.0	30.0	40.0	45.0	50.0	55.0	60.0	80.0
Distilled water, ml.	53.4	47.5	41.55	35.6	32.65	29.65	26.7	23.75	11.85
Total volume refluxed, ml.	100	100	100	100	100	100	100	100	100
pH of equilibrium mixture cooled to room temp. (chromic nitrate reactions)	3.42	3.21	3.06	3.05	3.02	2.97	2.96	2.91	2.87
Total volume to which equilibrium mixture is diluted, ml.	250	250	250	250	250	250	250	250	250
Volume taken for further dilution to 100 ml. to give $0.5 \times 10^{-4}$ M. based on original dye content, ml.	13.9	15.7	17.85	20.85	22.75	25.0	27.8	31.25	62.5
pH of final dilution (chromic nitrate reactions only)	5.08	4.52	4.33	4.20	4.11	4.08	4.00	3.94	3.54

minimal quantities of eluant were used and the fractions were concentrated for spectrophotometric examination.

In early work the sums of the optical density contributions of the fractions eluted differed by up to 10% from the optical densities of the original mixtures, although duplicate analyses agreed very well. By separate experiment, it was shown that dye and 1:1 complex could be quantitatively recovered separately. The loss was presumably of 2:1 complex. It was finally discovered that some of this remained, staining the lower three sections on the column, from which the complexes had been eluted with buffer. The amount remaining merely produced very slight darkening of the cellulose, only noticeable by comparison with a freshly made column, or on viewing the column along its length.

The staining could be removed by elution with water, gathering into a narrow violet band as the water displaced the buffer. On adding the optical density contribution of this final band, complete agreement was obtained over considerable wavelength ranges between the sums of the optical density contributions of the fractions and the original mixture curve (see Fig. 5 and 6).

Identity of this final fraction with the main 2:1 complex fraction was strongly suggested by the fact that their logarithmic density-wavelength curves were superimposable and by a similar recovery of dye on boiling with oxalic acid. In the figures and calculations, both these fractions have been included as  $\text{CrD}_2$ .

\* \* \*

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## EXPLANATORY PAPER ON MODERN THEORY

## Disperse Dyes on Hydrophobic Fibres

C. L. BIRD

Although disperse dyeing appears at first sight to be very different from the application of dyes which are readily soluble in water, closer study shows that the two processes are in fact similar. But disperse dyes are not ionised, and dyeing does not depend on the presence of salt-forming groups in the fibre molecule, so disperse dyeing systems are much easier to treat theoretically than the older systems. In spite of the absence of ionising sulfo groups in the dye molecule, disperse dyes are not completely insoluble in water. They diffuse into the fibre from a very dilute aqueous solution, which, as dye is removed from solution by the fibre, is replenished owing to the presence of fine particles of suspended dye. At equilibrium a linear relationship is found between dye on the fibre and dye in the bath, so the dyeing process resembles the partition of a solute between two immiscible solvents.

## INTRODUCTION

Research into the nature of dyeing processes arises primarily from curiosity—the desire to know how things work. But dyeing theory has practical value—both to the dye maker and to the dye user—as well as giving intellectual satisfaction.

A chauffeur-mechanic may not drive any better than an ordinary chauffeur, but he is far more resourceful when the car breaks down. Similarly, a technical dyer is more competent to face new problems than is the rule-of-thumb dyer. Nowadays a technical dyer needs to know a great many facts about fibres and dyes, and he requires a framework in which to assemble his facts in an orderly manner. This framework is provided by dyeing theory, which has a twofold purpose, viz. (a) to provide a picture of the dyeing process, and (b) to give numerical values to dyes so that their behaviour under any set of conditions can be predicted.

Dyeing research has been mainly concerned with three fundamental aspects, viz.—

- (1) The rate of diffusion of dye in the fibre under standard conditions
- (2) The affinity of dyes
- (3) The effect of varying temperature on (1) and (2).

The required data are usually obtained from carefully controlled dyeings at constant temperature, using purified fibres and purified dyes. From such dyeings we can obtain for various temperatures—

- (i) The rate of dyeing
- (ii) The ratio of dye on the fibre to dye in solution when the dyeing has reached equilibrium.

Rate of dyeing depends on many factors, e.g. the diameter of the fibres and the efficiency of stirring, neither of which is of fundamental significance. What is fundamental is the rate of diffusion of the dye in the fibre. This can be obtained from the rate of dyeing by means of the appropriate mathematical equation. These equations are often complicated and rather frightening to the beginner. But they should be regarded merely as tools. In a similar manner, many of us

use pH-meters without being able to explain just how they work, although we know what pH means. We shall see later how easy it is to determine diffusion coefficients from rate-of-dyeing experiments.

The affinity of a dye determines its distribution between fibre and dyebath at equilibrium, the higher the affinity for the fibre the greater being the degree of exhaustion. With ordinary water-soluble dyes the equilibrium is affected by the presence of acids and salts, but this is not so with disperse dyes, and the partition is a direct measure of affinity. Hence the theory of dyeing is much simpler with disperse dyes than with other classes of dyes.

In this paper an attempt is made to explain the present theory of disperse dyeing in a way which should be intelligible to most technical dyers. The more mathematical part, which is not essential to the main thesis, has been relegated to the appendix. In order to avoid overlapping with previous papers in this series<sup>1,2</sup>, not every aspect of dyeing theory has been covered. In particular, the effect of temperature on rate of diffusion—the activation energy of diffusion—has not been discussed.

## DISPERSE DYES

Cellulose acetate rayon, which came on the market shortly after the 1914–1918 war, may be regarded as the first of the hydrophobic (water-repellent) fibres. It is much less hydrophobic than, for example, Terylene and Orlon, but compared with the natural fibres and the cellulosic rayons it does not swell very much in water, and so will admit only small dye molecules. It has very little affinity for ordinary water-soluble acid dyes and direct dyes.

To be suitable for acetate rayon a dye must be soluble in organic esters and have a low affinity for water, i.e. its aqueous solubility must be low. Since satisfactory dyes were not originally available for dyeing acetate rayon, it was necessary to develop a special range, viz. the disperse dyes, which were later found to be suitable for nylon, Terylene, and Orlon. The type is illustrated by Dispersol Fast Orange G and Duranol Red 2B, which may be compared with the direct dye, Chlorazol Sky Blue FF—



(3) *Van der Waals Forces*

These are the forces of attraction which operate when molecules come close together. For example, they are responsible for holding together the sheets of carbon atoms found in graphite. They are weak forces, so it is necessary for a considerable area of two molecules to lie in contact in order to obtain appreciable cohesion. In cellulose the glucose rings present a flat surface on which the long flat molecules of direct dyes can lie. This form of attraction may operate to some extent with disperse dyes, especially with Terylene, where the benzene rings of the dye molecules may lie flat on the benzene rings which occur at intervals along the polymer chains.

It is possible that in all dyeing processes more than one type of linkage is involved.

## DIFFUSION IN THE FIBRE

Dyeing commences with adsorption of dye at the fibre surface, so in the early stages the concentration of dye is high in the surface layer and falls away rapidly as we pass from the surface to the centre of the fibre. When such a difference in concentration exists, diffusion will tend to take place in order to eliminate it. Hence dye adsorbed on the surface of a fibre will diffuse inwards until the fibre is uniformly coloured. The difference in concentration per unit distance is termed the *concentration gradient*.

The fibre may be regarded as a molecular sliver. With natural fibres and cellulosic rayons the sliver swells on wetting and the spaces between the long molecular chains are filled with water (except in the tightly packed crystalline regions). In these water-filled spaces dyes are free to diffuse. Since the dyes have affinity for the fibre, however, their free passage is hindered, as though they were continually sticking to the sides of the waterways and having to break loose again. The higher the affinity, the slower the progress. Fibre molecules are in a perpetual state of vibration owing to thermal agitation, and this causes the spaces between them to open and close. Dye molecules will be held up by narrow spaces and will have to wait for the space to widen before passing through.

Diffusion is much more rapid at high temperatures than at low temperatures. The dyes themselves become more active, and the spaces between the fibre molecules open and close more rapidly.

With very hydrophobic fibres such as Terylene there is no swelling in water and there is a negligible amount of water in the fibre. Hence diffusion cannot take place through water-filled spaces, and we must regard the fibre itself as a liquid—albeit a highly viscous one—in which non-ionic dyes are soluble and can diffuse. It is only when the temperature is raised above 100°C. that the fibre opens up sufficiently to admit dye molecules fairly freely. Alternatively, the fibre may be caused to swell by adding a swelling agent to a boiling dye-bath. Because there is so little swelling the dye molecules must be as small as possible.

Acetate rayon is an intermediate case. Slight swelling takes place in water, and disperse dyes probably diffuse in this fibre partly in aqueous

solution and partly by movement from site to site along the fibre molecules without any assistance from the water. Those dyes with the smallest molecular size diffuse most rapidly.

## MEASUREMENT OF DIFFUSION COEFFICIENTS

Dyeing with disperse dyes differs from other forms of dyeing in that, for nearly all the time, it is taking place from a dilute saturated solution which is in equilibrium with a saturated film of dye on the surface of the fibres. Except at high temperatures, i.e. above 100°C., the dye-bath remains saturated for almost all the time owing to the low aqueous solubility of disperse dyes, most of the dye being present in the form of a reserve of fine particles.

Provided that there is sufficient movement of the dye liquor to prevent the formation of a stagnant surface layer, the surface of the fibres will remain saturated as long as there is a reserve of suspended dye. Under these conditions the rate of dyeing is controlled by the rate of diffusion of dye in the fibre.

According to Fick's law—

$$\frac{\text{Rate}}{\text{Gradient}} = \text{Constant} \quad (i)$$

for the diffusion of any particular dye at a given temperature; i.e. the rate of diffusion of dye in the fibre is proportional to the gradient set up by the higher concentration of dye on the surface of the fibre.

To illustrate this relationship let us consider the case of a cyclist coasting down a hill. Suppose that his speed is 20 m.p.h. for a gradient of 1 in 20. Then, if Fick's law were obeyed, his speed for a gradient of 1 in 10 (2 in 20) would be 40 m.p.h. Some bicycles develop less friction than others and enable one to travel faster down a hill, but the difference is not very great. Dyes show a much greater variation, however. Thus, with disperse dyes the range of diffusion speeds in acetate rayon at 80°C. is about 100:1 for the same concentration gradient.

When a dye diffuses in a substrate such as cellulose acetate, diffusion will be of one of two types, viz. either—

- (a) Steady-state, or
- (b) Non-steady-state,

depending on the conditions.

(a) *Steady-state Diffusion*

Suppose that a disperse dye is diffusing through a 1-cm.<sup>2</sup> disc of cellulose acetate film from one side *AB*, and is being removed as soon as it emerges from the other side *CD*, as illustrated diagrammatically in Fig. 1. If the solution is kept saturated the concentration of dye on the surface of the film will be the fibre saturation value *EB*\*. The concentration at *CD* will be zero. The steepness of *ED* is the concentration gradient, which will not change so long as the solution is kept saturated and no dye is allowed to accumulate to the right of *CD*. This is steady-state diffusion.

\* The concentration of dye at the film surface (*EB*) will probably be 200–2000 times that of the saturated solution, which is therefore too small to be represented in Fig. 1.

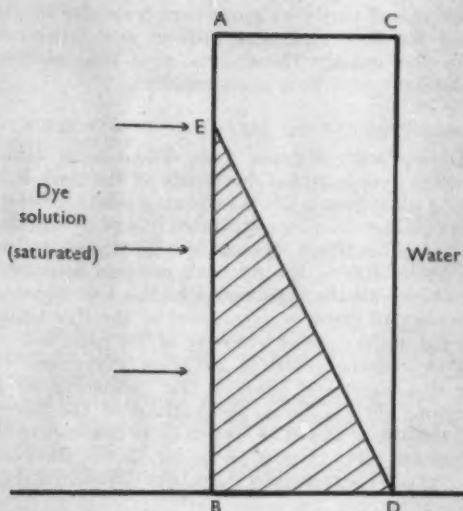


FIG. 1—Steady-state Diffusion

If we measure the rate at which dye is emerging from  $CD$  we obtain the *rate of diffusion*. The *concentration gradient* is obtained by first measuring the amount of dye in the film. From Fig. 1 it is clear that the average concentration so obtained is  $\frac{1}{2}EB$ . Multiplying by 2 therefore gives us  $EB$ , and on dividing this value by the thickness of the film ( $BD$ ) we obtain the concentration gradient. On dividing the rate of diffusion by the concentration gradient we obtain the constant in equation (i), which is known as the *diffusion coefficient* and is the rate of diffusion for unit concentration gradient.

**EXAMPLE—**Suppose that Duranol Red 2B is diffusing at  $80^{\circ}\text{C}$ . through a circular disc of cellulose acetate film of area  $15\text{ cm}^2$  and thickness  $0.001\text{ cm}$ . The rate of diffusion is found to be  $7.5 \times 10^{-8}\text{ g./sec.}$  and the amount of dye in the film  $0.0084\text{ g./cm}^2$ . Clearly, the amount of dye diffusing in a given time, and hence the rate of diffusion, will depend on the area of the disc, so the rate of diffusion must be corrected to give the value for  $1\text{ cm}^2$ , which in this case entails dividing by 15, giving  $0.5 \times 10^{-8}\text{ g./sec. per cm}^2$ . The concentration gradient is—

$$\frac{2 \times 0.0084}{0.001} = 16.8\text{ g./cm}^2\text{ per cm.}$$

and the diffusion coefficient is—

$$\frac{0.5 \times 10^{-8}}{16.8} = 3 \times 10^{-10}\text{ cm}^2/\text{sec.}$$

### (b) Non-steady-state Diffusion

In the dyeing of textile fibres, the concentration gradient is not constant, but decreases steadily throughout the dyeing. This is illustrated diagrammatically in Fig. 2 for a dyebath containing sufficient dye to saturate the fibre. In  $A$  the dye has not yet reached the centre, i.e. the fibre is "ring-dyed". In  $B$  the dye has reached the centre of the fibre, but the concentration at the centre is less than that at the surface. In  $C$  the fibre is in equilibrium with the bath; it is completely penetrated, diffusion has ceased, and the concentration gradient is zero.

Usually the amount of dye present is far from being sufficient to saturate the fibre at the end of the dyeing, and the course of events is as illustrated in Fig. 3. Stage  $A$  represents the point where the last particle of suspended dye has just dissolved and the *surface* of the fibre is still in equilibrium with a saturated solution of dye. At this point the bath is practically exhausted, but the fibre is only ring-dyed. Stage  $C$  shows the true equilibrium state with the fibre fully penetrated, and  $B$  is an intermediate stage.

Fig. 3 illustrates the course of events when diffusion into the fibre is slow; as when Terylene is dyed at  $100^{\circ}\text{C}$ . without any addition of swelling agent. Where diffusion into the fibre is much more

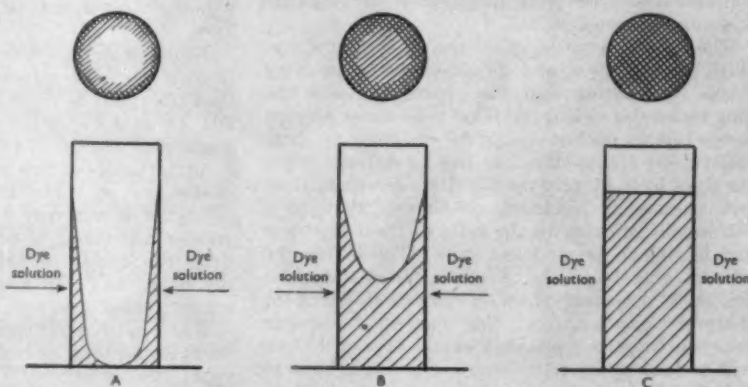


FIG. 2—Diffusion into a Fibre from a Bath containing an Excess of Dye

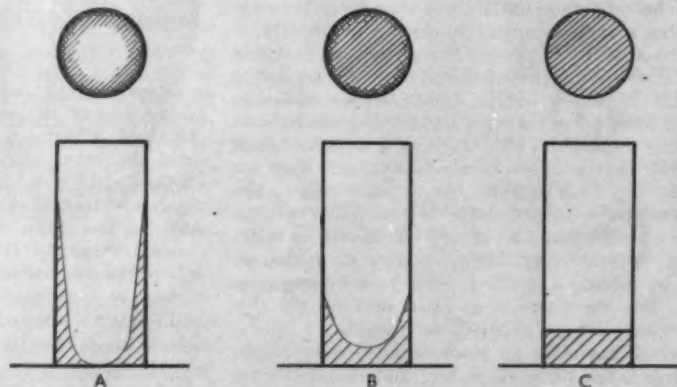


FIG. 3—Diffusion of Dye in a Fibre

rapid, as with acetate rayon and nylon, the dye particles may not dissolve sufficiently rapidly and the movement of dye liquor may be insufficient to maintain a saturated surface film. Under such conditions stage *B* will represent the point where maximum exhaustion (but not equilibrium) is reached.

The diffusion coefficient of a dye can readily be determined from rate-of-dyeing experiments provided that the fibre has a circular cross-section, and this method has been used with nylon and Terylene, the conditions being as in Fig. 2. All that is necessary is to find out (1) the amount of dye taken up at equilibrium ( $C_\infty$ ) and (2) the amount of dye taken up after dyeing for a short time ( $C_t$ ). The ratio of these two values is related to the diffusion coefficient  $D$ , the time  $t$ , and the square of the radius of the fibre  $r$ , i.e.—

$$\frac{C_t}{C_\infty} \text{ is related to } \frac{Dt}{r^2} \quad (\text{ii})$$

The actual equation is a complicated one, but solutions in the form of tables or graphs are available<sup>3</sup>, so the calculations present no difficulty.

**EXAMPLE (after Vickerstaff)**—Suppose that 43-denier, 17-filament nylon is dyed at 80°C. with commercial Duranol Red X3B 300. Since (a) the density of nylon is 1.14 g. per cm.<sup>3</sup>, (b) the denier is equal to the weight of 9000 metres of yarn, and (c)—

$$\text{Weight} = \text{Volume} \times \text{Density}$$

we have for each filament, on converting metres to centimetres—

$$\frac{43}{17} = \pi r^2 \times 9000 \times 100 \times 1.14$$

$$r^2 = \frac{43}{17 \times \pi \times 9000 \times 100 \times 1.14} = 8 \times 10^{-7} \text{ cm.}^2$$

It is found that the saturation value  $C_\infty$  is 7.5%, whilst after dyeing for 20 min. the nylon takes up 6.0% dye. Hence—

$$\frac{C_t}{C_\infty} = \frac{6.0}{7.5} = 0.8$$

From the tables<sup>3</sup> we find that this figure corresponds to a value of 0.215 for  $Dt/r^2$ . Hence—

$$D = \frac{0.215 \times 8 \times 10^{-7}}{20 \times 60} = 1.4 \times 10^{-10} \text{ cm.}^2/\text{sec.}$$

#### RATE OF DYEING

To the practical dyer one of the most important properties of a dye is its rate of dyeing, which is mainly dependent on the rate at which the dye diffuses in the fibre. Equation (i) shows that—

$$\text{Rate (of diffusion)} = \text{Constant} \times \text{Gradient} \quad (\text{i})$$

The constant in this equation is the diffusion coefficient. The gradient is not constant, but decreases as dyeing proceeds, and is proportional to the saturation value. Hence the rate of dyeing of a particular disperse dye will depend on—

- The diffusion coefficient, and
- The saturation value.

A dye may have a high diffusion coefficient and yet be relatively slow-dyeing, because it has a low saturation value; whilst a low diffusion coefficient

may be counterbalanced to some extent by a high saturation value.

#### THE EQUILIBRIUM STATE

If a series of dyeings is carried out to equilibrium with one of the hydrophobic fibres and increasing amounts of a pure disperse dye, and the amounts of dye on the fibre ( $[D]_f$ ) and in the residual solution ( $[D]_a$ ) are then measured, a curve can be plotted, as shown in Fig. 4 for Terylene and Duranol Red 2B at two different temperatures. The same straight lines are obtained if dyed Terylene is treated with water at the two temperatures until the material is in equilibrium with a solution of desorbed dye. Thus the equilibrium is a reversible one.

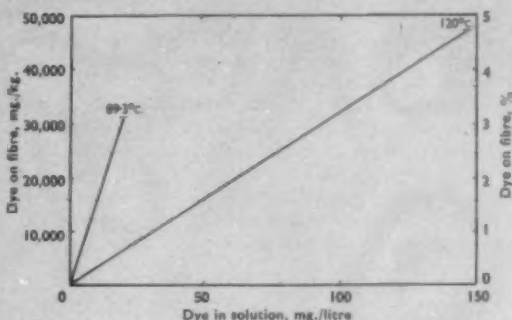


FIG. 4—Equilibrium Isotherms for Duranol Red 2B and Terylene (after Schuler and Remington)

The point at the top of each line in Fig. 4 gives the solubility of the dye in the fibre and in water for the particular temperature. The actual values are given in Table I.

TABLE I

	89.3°C.	120°C.
Solubility in Terylene (mg./kg.)	31,300	47,300
Solubility in water (mg./litre)	20	147
Ratio of solubilities (partition coefficient)	1,570	320

The ratio of the solubilities is known as the *partition coefficient*  $K$ , and since the relationship  $[D]_f/[D]_a$  is linear (Fig. 4),  $K$  is constant right up to the saturation point for any particular temperature, i.e.—

$$\begin{aligned} \frac{[D]_f}{[D]_a} &= \frac{\text{Fibre saturation value}}{\text{Aqueous solubility}} \\ &= \text{Constant} = \text{Partition coefficient} = K \quad (\text{iii}) \end{aligned}$$

It is clear from this equation that, if two of the variables are known, the third can be calculated. Thus, if the partition coefficient and the aqueous solubility are known, the solubility in the fibre (fibre saturation value) can be calculated.

Table I shows that raising the temperature increases the solubility of the dye in both water and the fibre, but the aqueous solubility is increased much more than the solubility in the fibre. Hence the partition coefficient decreases with rising temperature, as illustrated in Fig. 5. This means that, although it is possible to get more dye to go on to the fibre by raising the temperature, the equilibrium exhaustion of the dyebath will be lower.

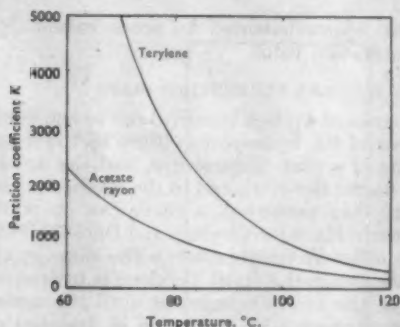


FIG. 5—Effect of Temperature on the Partition Coefficient of Duranol Red 2B on Acetate Rayon and Terylene

The amount of dye present is usually insufficient to saturate the fibre, so it may be found advantageous in practice to bring the dyeing to equilibrium rapidly at the highest permissible temperature, and then cool the bath so as to take advantage of the higher partition coefficient (greater exhaustion) at lower temperatures.

If the partition coefficient  $K$  and the liquor ratio  $L$  are known, the percentage exhaustion at equilibrium is given by the formula—

$$\text{Exhaustion (\%)} = \frac{K \times 100}{K + L} \quad (\text{iv})$$

EXAMPLE—Suppose that the experiments illustrated by Fig. 4 referred to Terylene cheeses and that the liquor ratio was 25 : 1. Since the partition coefficient at 120°C. is 320 (Table I), the percentage exhaustion would be—

$$\frac{320 \times 100}{320 + 25} = 93\%$$

At 89.3°C. the exhaustion would be 98%, but a very long time would be required to reach equilibrium on Terylene at this temperature.

Alternatively, if the percentage exhaustion is known, the value of  $K$  can be calculated. In practice, however, there is a danger that the dye-bath may contain some relatively large dye particles which dissolve extremely slowly and so give a false equilibrium. Much more reliable results are obtained if equilibrium is reached by desorbing the dye from dyed yarn.

It is evident from Fig. 5 that the partition coefficient is higher for Terylene than for acetate rayon, at all temperatures. Hence from equation (iii) the fibre saturation value at any particular temperature must be greater for Terylene than for acetate rayon. Whilst this is true for Duranol Red 2B, it is not generally true. The available data show that the fibre saturation values are usually about twice as great on acetate rayon as on nylon, whilst the values for Terylene may be less than those of nylon or greater than those of acetate rayon. Orlon has a low affinity for disperse dyes, and, in consequence, the fibre saturation values are very low.

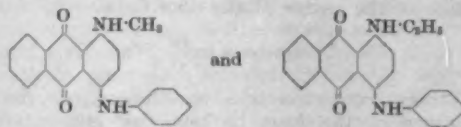
Fibre saturation values are of considerable practical importance. If the saturation value is low the dye will not "build up" well. Some blue disperse dyes, for example, will build up to a navy blue if sufficient dye is present, but others will not

because the saturation value is soon reached. From equation (iii) it is seen that—

$$\text{Fibre saturation value} = \text{Partition coefficient} \times \text{Aqueous solubility} \quad (\text{v})$$

With acetate rayon it is found that, in general, the affinity of the dye, as shown by the partition coefficient, decreases as the aqueous solubility increases. On the other hand, with some dyes of very low aqueous solubility, the partition coefficient is not sufficiently high to make up for the low aqueous solubility, so these dyes do not build up well.

An alternative method of ensuring good build-up, which is particularly useful with Orlon, is to use a suitable mixture of two (or more) dyes. Some disperse dyes, e.g.—



form mixed crystals, the mixture showing little improvement in build-up. With other mixtures, where the components are dissimilar, the dyes build up separately, and the saturation value is the sum of the individual values.

#### EFFECT OF TEMPERATURE

The effect of temperature on the aqueous solubility of disperse dyes is illustrated in Fig. 6, for Duranol Red 2B. It is seen that the aqueous solubility is very low up to about 60°C., and then increases rapidly. At 120°C. all the suspended dye dissolves to give a clear solution at the start of dyeing with shades up to medium depth, provided that the liquor ratio is not too small.

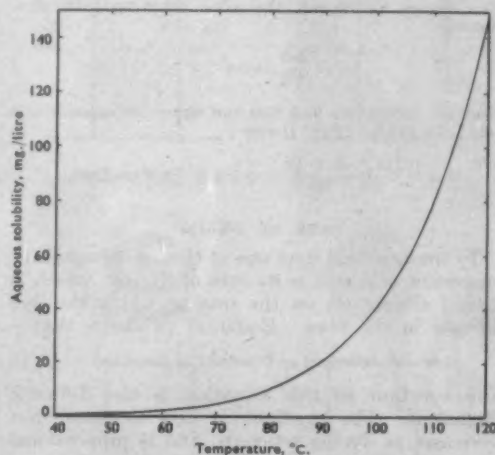


FIG. 6—Effect of Temperature on the Aqueous Solubility of Duranol Red 2B

EXAMPLE—Suppose that 100 lb. of Terylene cheeses are to be dyed at 120°C. in 25 vol. with 1.5% Duranol Red 2B 300. If the strength of the commercial dye is 20%, 0.3 lb. of dye is present in 2,500 lb. of water. Hence the concentration is 0.012%, which is less than the solubility of the dye at 120°C., viz. 0.0147% (see Table I).

A curve of the type illustrated in Fig. 6 denotes a logarithmic relationship, and if the logarithm of the aqueous solubility (Table I) is plotted against the reciprocal of the absolute temperature, a straight line is obtained, as shown in Fig. 7. By extending the curve, as shown by the broken lines in Fig. 7, we can obtain the aqueous solubility at other temperatures without the need for actual measurement.

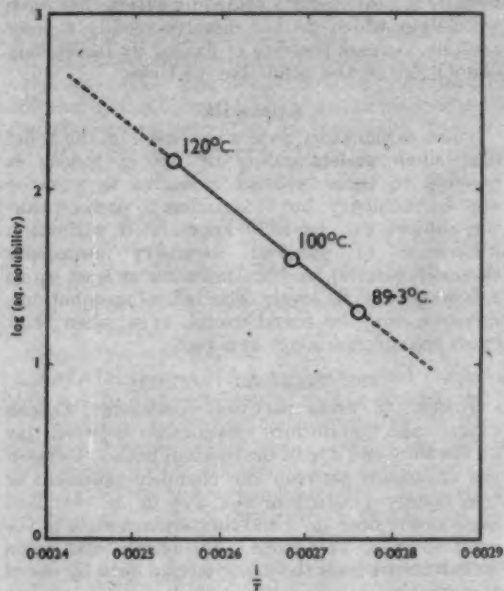


Fig. 7—Duranul Red 2B: Linear Relationship between the Logarithm of the Aqueous Solubility and the Reciprocal of the Absolute Temperature

**EXAMPLE**—Suppose that we wish to know the solubility at 140°C., which is  $140 + 273 = 413^\circ\text{Absolute}$ . The reciprocal is  $1/413 = 0.00242$ . From the graph the corresponding logarithmic solubility is 2.66; hence the aqueous solubility is 460 mg./litre.

In a similar manner we can plot the logarithm of the solubility in the fibre (Table I) against the reciprocal of the absolute temperature and, by extending the straight line so obtained (Fig. 8), calculate the solubility in the fibre at other temperatures.

A plot of the logarithm of the partition coefficient against  $1/T$  also gives a straight line (Fig. 9). In this case the slope is reversed, because  $K$  decreases with rise in temperature. Again, by extending the

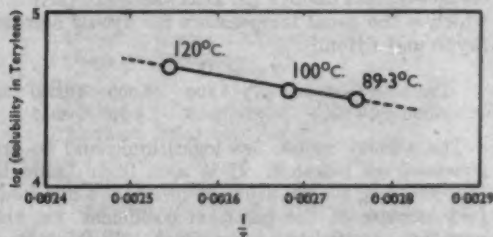


Fig. 8—Duranul Red 2B: Linear Relationship between the Logarithm of the Solubility in the Fibre and the Reciprocal of the Absolute Temperature

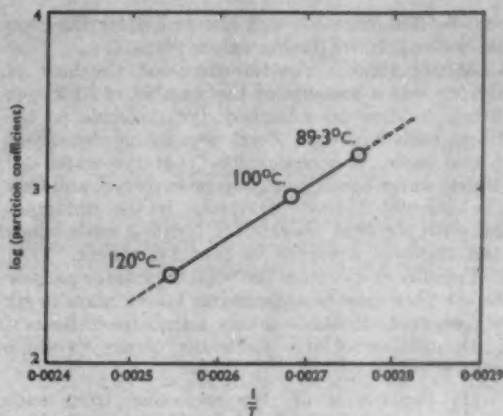


Fig. 9—Duranul Red 2B: Linear Relationship between the Logarithm of the Partition Coefficient and the Reciprocal of the Absolute Temperature

line we can obtain values of  $\log K$  for other temperatures.

#### HEATS OF SOLUTION AND OF DYEING

The slope of the straight line in Fig. 7 is a measure of the effect of temperature on the aqueous solubility, the greater the slope the greater being the temperature effect. The heat of solution in water  $\Delta H^\circ_{\text{H}_2\text{O}}$  can be obtained in the following way. Taking any two convenient points, e.g. the values for the logarithm of the aqueous solubility at 120° and 89.3°C. in Fig. 7, we have, on subtracting—

$$2.167 - 1.301 = 0.866$$

This figure is multiplied by a constant  $R$  (1.99 calories/mole per degree centigrade) and the factor (2.303) for converting common logarithms to natural logarithms, and divided by the difference between the corresponding two values for  $1/T$ , i.e.—

$$0.002761 - 0.002545 = 0.000216$$

Hence—

$$\Delta H^\circ_{\text{H}_2\text{O}} = \frac{0.866 \times 1.99 \times 2.303}{0.000216} = 18,400 \text{ cal./mole}$$

Similarly we can obtain the heat of solution in the fibre  $\Delta H^\circ_{\text{Fibre}}$ : whilst the slope of  $\log K$  against  $1/T$  enables us to obtain the heat of dyeing  $\Delta H^\circ_{\text{Dye}}$ . The relationship between these three values is—

$$(\text{Heat of solution in fibre}) - (\text{Heat of solution in water}) = \text{Heat of dyeing}$$

or

$$\Delta H^\circ_{\text{Fibre}} - \Delta H^\circ_{\text{H}_2\text{O}} = \Delta H^\circ_{\text{Dye}} \quad (\text{vi})$$

For Terylene and Duranul Red 2B we have—

$$\Delta H^\circ_{\text{Dye}} = 3.7 - 18.4 = -14.7 \text{ kcal./mole}$$

Since the heat of aqueous solution is always greater than the heat of solution in the fibre (as shown by the greater slope), the heat of dyeing is always negative. This is illustrated by the reverse slope in Fig. 9 compared with the slopes in Fig. 7

and 8. The negative sign also indicates that heat is evolved when dyeing takes place.

At one time it was thought that the heat of dyeing was a measure of the number of hydrogen bonds holding an adsorbed dye molecule to the fibre, each hydrogen bond accounting for about 5 kcal./mole. It is now realised that dye-water and water-water bonds must also be involved, and that the calculated heat of dyeing is the difference between the heat liberated in forming some bonds and the heat absorbed in breaking others.

Transfer of dye from the solid crystalline particle to the fibre may be regarded as taking place in six stages, each of which entails a transfer of heat.

Dissolution of dye in water occurs in three stages, viz.—

- (1) Separation of dye molecules from each other in the crystal. (Heat absorbed.)
- (2) Separation of water molecules from each other to make room for the dye. (Heat absorbed.)
- (3) Formation of links between dye molecules and water molecules. (Heat liberated.)

The heat absorbed in stages (1) and (2) is greater than the heat liberated in stage (3), so the net heat of aqueous solution ( $\Delta H^\circ_{H_2O}$ ) is positive. Hence dissolution of dye is promoted by heating.

When dye is adsorbed from solution by the fibre, three stages are again involved, viz.—

- (4) Breakdown of links between dye molecules and water molecules. (Heat absorbed.)
- (5) Re-formation of bonds between water molecules. (Heat liberated.)
- (6) Formation of links between dye molecules and the fibre. (Heat liberated.)

Stages (4) and (5) are the opposite of stages (3) and (2); the heat change has the same magnitude, but is of opposite sign. Since the heat liberated in stages (5) and (6) is greater than the heat absorbed in stage (4), the net heat of dyeing ( $\Delta H^\circ_{Dye}$ ) is negative. Hence if the temperature of a dyebath which has reached equilibrium is raised, dye is transferred from the fibre to the bath.

#### THE DISPERSING AGENT

Although it is possible to apply disperse dyes without any addition to the dyebath other than the dye itself, a dispersing agent is always added in practice. This has a stabilising effect on the fine aqueous dispersion, since it combines with the minute dye particles and prevents them from coalescing and separating out in the form of aggregates. Besides attracting each other, the dye particles are attracted by the fibre in the absence of dispersing agent (as can be seen under the microscope), and particles of dye adsorbed on the surface in this way give rise to dyeings which have poor fastness to rubbing.

The dispersing agents added to the dyebath—to the extent of 1–2 g./litre—are all detergents or closely related compounds. Such substances possess the power of "solubilising" compounds which are virtually insoluble or are only very slightly soluble in water. This is due to their

property of forming aggregates or micelles. Soap, for example, exists in solution partly as sodium ions and fatty acid ions and partly as micelles. The micelles may be regarded as minute droplets of hydrocarbon in which disperse dyes can dissolve.

Hence the dispersing agent competes with the fibre for the dye. It increases the amount of dye in solution and so reduces the partition coefficient and the percentage exhaustion of the dyebath. Usually it also exerts a retarding action, but with some dyes which do not dissolve readily it may actually increase the rate of dyeing by facilitating dissolution of the solid dye particles.

#### Appendix

These explanatory papers are based on the belief that some understanding of dyeing theory is possible to those without a degree in pure or applied chemistry, but it is useless to pretend that the subject can be fully appreciated without a knowledge of physical chemistry (especially thermodynamics), and mathematics at least up to Advanced G.C.E. level. The following equations, however, may be found useful, even when their exact significance is not apparent.

#### THE STANDARD AFFINITY

Instead of using partition coefficients we can express the equilibrium relationship between dye on the fibre and dye in the residual bath in terms of the difference between the chemical potential or free energy ( $-\Delta\mu^\circ$ ) of the dye in its standard state in the fibre ( $\mu_f^\circ$ ) and its standard state in the solution ( $\mu_s^\circ$ ). This value is the *affinity*, and it can be calculated from the experimental data by use of the equation—

$$-\Delta\mu^\circ = RT \ln \frac{[D]_f}{[D]_s} = 2.303 RT \log \frac{[D]_f}{[D]_s} \quad (\text{vii})$$

where  $R$  is the gas constant and  $T$  is the absolute temperature. The amounts of dye on the fibre  $[D]_f$  and in the solution  $[D]_s$  are expressed as moles per kilogram of absolutely dry fibre and moles per litre, respectively. With disperse dyes  $[D]_f/[D]_s$  is the partition coefficient.

Referring to Table I we see that the partition coefficient for Duranol Red 2B distributed between Terylene and water is 320 at 120°C. (393°A.). Hence the affinity at 120°C. is seen from equation (vii) to be—

$$\begin{aligned} -\Delta\mu^\circ &= 2.303 \times 1.99 \times 393 \times \log 320 \\ &= 4308 \text{ calories per mole of dye} \end{aligned}$$

Table II shows the relationship between partition coefficient and affinity (in kcal.) at 80°C. (353°A.), which is the usual temperature for dyeing acetate rayon and nylon.

TABLE II

Partition coefficient ( $K$ )	100	1,000	10,000
Affinity ( $-\Delta\mu^\circ$ )	3.23	4.85	6.47

The affinity values are logarithmic and so are squeezed up together. It is seen from Table II that doubling the affinity corresponds to a hundred-fold increase in the partition coefficient, i.e. the partition coefficient is squared. With water-soluble dyes  $K$  is not constant, so affinities (which are independent of concentration) have to be used;

but with disperse dyes one can use partition coefficients, which are preferable, as their significance is readily appreciated.

#### HEATS OF SOLUTION AND HEAT OF DYEING

Instead of the graphical method, it may be more convenient to use the following equation in order to obtain the heat of aqueous solution—

$$\Delta H^\circ_{H_2O} = \frac{RT_1T_2}{T_1 - T_2} \ln \frac{\text{Sol}T_1}{\text{Sol}T_2} \quad (\text{viii})$$

where  $R$  is the gas constant, and  $T_1$  and  $T_2$  are the absolute temperatures corresponding to two solubilities  $\text{Sol}T_1$  and  $\text{Sol}T_2$ . Similarly, the heat of solution in the fibre ( $\Delta H^\circ_{\text{fibre}}$ ) can be determined if two fibre-solubility values are available. In order to obtain the heat of dyeing ( $\Delta H^\circ_{\text{Dye}}$ ), two partition

coefficients,  $K_{T_1}$  and  $K_{T_2}$ , are substituted for  $\text{Sol}T_1$  and  $\text{Sol}T_2$  in equation (viii). Thus, using the partition coefficients in Table I for 89.3°C. (362.3°C.) and 120°C. (393°C.), we have—

$$\begin{aligned} \Delta H^\circ_{\text{Dye}} &= \frac{1.99 \times 362.3 \times 393}{362.3 - 393} \times 2.303 \times \log \frac{1570}{320} \\ &= -14,700 \text{ calories per mole} \end{aligned}$$

The author has received a great deal of help and advice with regard to this paper from Mr. H. C. Olpin, Dr. L. Peters, and Dr. C. B. Stevens.

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- Peters, L., *ibid.*, **71**, 174 (April), 726 (Dec. 1955).
- Vickerstaff, T., *The Physical Chemistry of Dyeing* (London: Oliver & Boyd, 2nd edition 1954), pp. 132–133.

## CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

### Forlion—A New Polyamide Fibre

Your *Journal* (*J.S.D.C.*, **72**, 242 (May 1956)) and others have reported the manufacture of a "new polyamide fibre, formed by polymerisation from goat's milk by the process of Schlak and Kleine".

This report is too good to pass by: it is the result of a minor translation howler. *Angewandte Chemie* of 7th April 1956 has stated that the report concerning a new polyamide fibre from goat's milk originated in the *Neue Zürcher Zeitung* of 1st

January 1956. The German journal comments: "Caprolactam, from which the fibre is made, should not have been translated anyway by 'Ziegenmilch' [nanny-goat's milk] but by *Bocksmilch* [billy-goat's milk], because *capro* means the male of the species!"

R. M. LODGE

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## Notes

### Meetings of Council and Committees

#### June

Council—6th

Finance and General Purposes—6th

Colour Index Editorial Panel—8th and 21st

Publications—19th

Diplomas—13th

Fastness Tests Co-ordinating Committee—27th

Perkin Fund Advisory Committee—18th

### Election of Fellows

At the meeting of Council held on 4th July 1956 the following were elected Fellows of the Society—

Arved Datyner

(Leicester; Senior Lecturer in Dyeing, Leicester College of Technology)

Clifford Paine

(London; Development Director, Imperial Chemical Industries Ltd.)

Harry Palfreeman

(Montreal; Technical Adviser on Dyeing and Finishing, Courtaulds (Canada) Ltd.)

George Robinson

(Kidderminster; Chief Chemist, Carpet Trades Ltd.)

Walter Stanley Willson

(Pontypool; Senior Technical Service Representative, British Nylon Spinners Ltd.)

Reginald Frank York

(Billerica, Essex; Assistant Manager, Technical Development, Howards of Ilford Ltd.).

### Annual Meeting of Section Officers

The Annual Meeting of Chairmen and Honorary Secretaries of Sections was held at the Midland Hotel, Manchester, on Friday, 27th April 1956, under the chairmanship of Mr. L. M. Wood (Honorary Secretary of the Society). Among the topics discussed were membership cards, projection of slides at lectures, election of new members of the Society, fifty-years membership, meeting notices and the clashing of meetings, lecture titles, and the comprehensibility of papers in the *Journal*.

### Reports of Sections for the Session 1955–1956

#### Huddersfield Section

Once again we have to report a successful session. We have had seven lectures in all. These have been well attended, in spite of the weather and a change of meeting place half way through

the session. As in previous years we held a joint meeting with the Huddersfield Textile Society, when Mr. N. M. Mims gave a very good lecture on *The Finishing of Terylene and Terylene-Wool Blended Fabrics*. Great interest was shown in this lecture. Our Joint Lecture with the Huddersfield Section of the Royal Institute of Chemistry took place in January, when Professor J. B. Speakman gave a very full and interesting talk on *Recent Research on Wool*.

To mark the Perkin Centenary an exhibition was held in March. An excellent show was staged by Imperial Chemical Industries Ltd. at the Huddersfield Technical College as part of the College Exhibition, and was kept on an additional two days for the benefit of our members. Several exhibits were provided also by the Dyeing Department of the College, and a sample of Mauveine produced at the Greenford Green Works at the commencement of commercial production could be seen.

During the session members of the Section have visited Samuel Fox & Co. Ltd., the producers of stainless steel at Stocksbridge, near Sheffield, which proved very interesting and educational.

On 25th April 1956 thirty of our members visited Messrs. Stevensons (Dyers) Ltd. at Ambergate, Derbyshire. This was a most interesting and enjoyable day, and the visit was greatly appreciated by all who attended.

This report cannot be concluded without expressing appreciation to our Chairman and all members of the Committee, without whose help the session would not have been the success it has been.

HORACE TURNER

*Honorary Secretary, Huddersfield Section*

#### LONDON SECTION

All the technical lectures held during the session have, with one exception, which was probably due to bad weather, been well supported. Six lectures have been given at the Royal Society rooms, one jointly with the London Centre of the Guild of Dyers and Cleaners. In addition a joint meeting has been held at the Chemical Society rooms with the London Section of the Textile Institute. A meeting was again held at Luton and was well supported by local members.

The Annual Dinner and Dance was held at the Waldorf Hotel, and again proved to be a popular attraction.

The award of the Society's Silver Medal to Dr. H. W. Ellis for valuable services to the Society as Honorary Secretary of the London Section from 1938 to 1954 has given much satisfaction to members of the Section.

Unfortunately, owing to the illness of the lecturer, Mr. James Laver, it has not been possible to present the Fourth London Lecture this session, and it is now hoped that it will be given on 19th October 1956.

The Section's contribution to the Perkin Centenary Celebrations consists of a Prize Essay Competition and also a Dinner and Ball held on the Thursday (May 10th) of the week of the

celebrations in London. The essay competition has been made possible by the generosity of an anonymous donor. Details have been widely published in the technical press, and a good response is expected. The Dinner and Ball attracted members of other Sections of the Society, and members of other participating societies, who were in London for the celebrations. We were pleased to welcome as guests the President of the Society, the President of the Royal Institute of Chemistry, and the Chairman of the Association of British Chemical Manufacturers.

The Section's membership has continued to increase steadily, and has just passed the 200 figure, fifteen new members joining during the session.

The Annual General Meeting and Dinner concluded the year's activities.

H. W. TAYLOR

*Honorary Secretary, London Section*

#### MANCHESTER SECTION

Ten meetings have been held in Manchester during the session and one external joint meeting with the Textile Institute in Leek. Although the attendance has not been high at any meeting, it is encouraging to record an increase over the attendances for the previous session. If the trend continues during the next session, the Committee will be much more satisfied with the support of the Sectional meetings.

This year the Symposium, on *The Second Hundred Years*, occupied the whole day, with an attendance of 120. Four lecturers gave excellent papers on dyeing, the future of new fibres, printing, and finishing. The Section held its Annual Dance in the Midland Hotel, with a record number of members and guests attending. The opinion is held that this was the most successful dance held by the Section. Although the price of the tickets had to be increased as a result of the cost of accommodation in the Midland Hotel, it is felt that the success of the evening was partly due to the very satisfactory service obtained.

During 1956 the Section celebrated the discovery of Mauveine by W. H. Perkin. A very successful exhibition was held for a fortnight in the Central Library to show the importance of Perkin's discovery and its influence on the dye and textile industries in Manchester. In addition, the discovery was made the theme of the One-day Symposium and the Annual Dance. It was interesting to see many well known members of the Section displaying mauve nylon handkerchiefs, dyed with Mauveine, during this social event.

The Knecht Memorial Prize for 1954-1955 was awarded to Mr. P. Brocklehurst, of the Manchester College of Technology, who obtained a First Class Honours Degree in Applied Chemistry (Colouring Matters).

High attendances were recorded at the Junior Branch meetings, but unfortunately two meetings had to be cancelled owing to late cancellations by the lecturers, who had other urgent Society business to attend to.

The Committee records with regret the retirement of two members, Mr. J. G. Grundy and Dr. J. M. Preston, by whom the Manchester Section has been well served. The Section must record its thanks to Professor R. H. Peters and the Manchester College of Technology for the use of accommodation for Committee meetings, and to the Manchester Corporation Library Committee for the use of the Exhibition Hall for the Perkin Exhibition.

Dr. P. W. Cunliffe is to be congratulated on the award of a Bar to his Gold Medal; and Mr. J. G. Grundy, Mr. K. McLaren, and Dr. M. R. Probert on the award of the Society's Silver Medal.

The membership of the Section increased by 51 from 652 to 703 members.

H. R. HADFIELD

*Honorary Secretary, Manchester Section*

#### MIDLANDS SECTION

A most gratifying feature of the session just ended has been the much increased attendances at lectures. Nine in all were held (Loughborough 3, Leicester 2, Nottingham 2, Derby 1, and Kidderminster 1), and the average attendance (99) rose over 50%. A Ladies' Evening Dinner Dance and a Dinner were also held; both were highly successful. The numerical statement of membership is as follows: new members 24, transferred to Section 6, transferred from Section 2, resignations 7, death 1—an increase in membership of 20.

At the Annual General Meeting of the Section the Chairman, Mr. C. A. Mills (who has been appointed for a further year of office) referred to the enthusiastic support he had been given by the other officers and by the committee. Midlands members had also played a good part in the general work of the Society. On the Council, we had as elected members: Messrs. Cluley, Tyler, Kershaw, and Edwards (Vice-president). The last two gentlemen now retired by rota, and he paid special tribute to them for their representation on the Finance and General Purposes Committee and the Diplomas Committee respectively. On the Diplomas Committee we were also represented by Messrs. Penn, Tyler, and Olpin, while Mr. Olpin also served on the Publications Committee.

Those responsible for preparing students for the Associateship of the Society had drawn attention to the lack of textbooks of a practical character. The Section had therefore suggested to the Publications Committee that the Society sponsor monographs to cover certain specific subjects. At the invitation of the Publications Committee, Dr. A. Dwyer, assisted by Mr. A. W. Carpenter, had written a monograph on *The Dyeing and Finishing of Half-hose*; the manuscript had now been submitted for approval.

The Chairman regretted to inform the meeting that Mr. Carpenter had signified his intention to retire from the Secretaryship in twelve months' time. To ensure continuity, Mr. J. Rayment was appointed Assistant Secretary by the meeting, it being anticipated that he would be Mr. Carpenter's successor in 1957. As a mark of appreciation, Mr. Carpenter was appointed Vice-chairman, with a

view to taking over the Chair during Mr. Rayment's first period of full office.

In the ballot for the Committee, Messrs. A. S. Cluley, D. B. McPherson, H. C. Olpin, and A. G. Tyler, retiring members, were re-elected, together with Mr. A. P. Kershaw.

We have pleasure in reporting that at the Annual General Meeting of the Society Mr. W. Penn was elected a Vice-president for the full period of six years, while Mr. W. L. Langton became an Ordinary Member of Council.

We regret to record the death of Mr. L. Mills, of Northampton.

A. W. CARPENTER

*Honorary Secretary, Midlands Section*

#### NORTHERN IRELAND SECTION

The symposium held at Portrush in September 1955 was the highlight of this session. It was a great honour to us that Council sanctioned the holding of this function in Northern Ireland, and we believe that a great deal of its success was due to the excellent selection of lectures organised by the small Papers Subcommittee working under the chairmanship of Mr. N. Hamer. We, on this side of the Channel, took much pleasure in dealing with the social and general arrangements, and we hope that sometime in the future we may have the privilege of having another symposium in Northern Ireland.

Other activities of the Section during the session were as follows—

LECTURES—Seven lecture meetings were held, two of these being joint functions with the Textile Institute. All were well attended.

COMMITTEES—Six symposium committee and five ordinary committee meetings were held.

ANNUAL DINNER—Following our Annual General Meeting on 27th March 1956 we had a good attendance at our Annual Dinner. Chief guests were our President, Mr. Fred Smith, Mr. D. A. Derrett-Smith, and Mr. J. Howard.

ANNUAL DINNER DANCE—On 17th February 1956 we had 180 members and friends at this function, which was very enjoyable.

ANNUAL GOLF TOURNAMENT—This was held in June 1955, at Massereene Golf Club, when 36 members took part in the competition.

Reviewing our general activities, we feel that, under the very capable chairmanship of Mr. R. J. G. Reid, we have had our most successful session, and we look forward to the future with confidence.

JAMES PORTER

*Honorary Secretary, Northern Ireland Section*

#### SCOTTISH SECTION

The 1955-1956 session has again been very successful and enjoyable. A total of ten ordinary meetings were held—nine in Glasgow and one in Galashiels—and attendance was remarkably good. In addition, the Thirteenth Mercer Lecture was held in Glasgow on 13th April 1956, and was very well supported and thoroughly enjoyed. We were particularly pleased to welcome Mr. Fred Smith and Mr. L. Morton Wood, who were paying their first official visit to our Section as President and Honorary Secretary respectively.

Sectional membership is much the same as at this time last year. Although we enrolled 14 new members during the session, these have been offset by removals, resignations, etc.

At the Annual General Meeting of the Section, Messrs. R. D. Alexander, H. R. Crone, C. H. Giles, and P. Schwarzenbach retired from the Committee by rota, and, as no other nominations had been received, these gentlemen were re-elected.

Mr. J. G. B. McCallum, former Vice-chairman, is now residing in Hawick and, in view of the difficulty of attending meetings with regularity, has asked that his name be withdrawn as nominee for Chairman, a decision that has been received with regret by the Committee. At the A.G.M. (1956) Mr. C. D. Smith was elected Chairman, and Mr. W. G. B. Grant became Vice-chairman.

In connection with local arrangements to commemorate the Perkin Centenary, a special lecture by Professor J. Read, on *The Life and Work of Perkin*, has been arranged for Tuesday, October 16th, and a Dinner-Dance for Friday, 28th September 1956.

Arrangements are in hand for the 1956-1957 session, and the Committee hopes to present a programme with practical appeal to all members.

F. ATACK

*Honorary Secretary, Scottish Section*

#### WEST RIDING SECTION

Under the Chairmanship of Mr. R. K. Fourness, the Section has held eight lectures on a wide range of subjects, which included the newer man-made fibres and the quality aspect of colour in textiles generally, from the point of view of the consumer. Of these meetings, six were held in Bradford and two in Leeds.

Three other Section events—the Supper and Smoker, the Ladies' Evening, and a lecture meeting to which ladies were invited—were completely successful, owing to the work of our Social Secretaries, Mr. J. M. Goodall and Mr. R. Hullah.

Average attendance at lecture meetings was 56, which compares favourably with previous years.

Unfortunately, this year the Competition for West Riding Juniors, based on the Dyers and Finishers' Association annual award of £10 10s 0d, has not been well supported by Junior Members, only one paper being offered.

The Section Committee has met on five occasions.

G. E. STYAN

*Honorary Secretary, West Riding Section*

#### Annual Golf Competition

The fourth Annual Golf Competition for the Goodall Trophy was held on Sunday, 3rd June

1956, on the course of the Leek Golf Club, Birchall, Leek, Staffordshire.

There was a record entry of thirty-four, but five had to withdraw at the last moment. The remaining twenty-nine (still ten more than the previous best entry) enjoyed excellent weather and a very pleasant game followed by a meal and the usual celebrations.

The Trophy was won by Mr. A. N. Russell, score 82 - 17 = 70. Four players—Messrs. Hepworth, Taylor, Newton, and Lord—tied for the second prize (presented to Mr. J. A. Hepworth on the best score for the last nine holes). The prize for the best gross score was won by Mr. R. D. Calvert.

The President was unable to attend owing to a prior engagement, and the prizes were presented to the winners by the Immediate Past President (Mr. Fred Smith).

#### International Fastness Label *Felisol*

The 'International Association for the Fastness Label *Felisol*' was founded in Zürich on 15th March 1956. Its object is to promote interest in textiles of high fastness to light, washing, and weathering, and permission to use the trade-mark *Felisol* will be granted to firms applying certain recommended dyes, in a prescribed manner, to cotton, regenerated cellulose, linen, hemp, and ramie. The Association is equally representative of Continental dyemakers (BASF, FBy, CFM, FH, Fran, ACNA, Ciba, Gy, S, DH, and Rohner) and Continental dye users (Association Internationale de la Teinture Textile, Associazione Cotoniera Italiana, Vereeniging van Loonveredelingsbedrijven voor de Textielindustrie, and Verband der Schweizerischen Textilveredelungsindustrie). Discussions are in progress with British and American dyemaking firms.

The *Felisol* label is to be employed first in Holland, Italy, and Switzerland. The headquarters of the Association are in Zürich. The President is Francis Brichet (Basle), and H. B. Nowak (Frankfurt-am-Main) and G. Artom (Milan) are Vice-presidents.

#### Light Fastness Testing at Aigues-Mortes

A 'station d'insolation' has been established at Aigues-Mortes in the South of France for testing the fastness of patterns to sunlight. Patterns must be forwarded in special envelopes and accompanied by special forms. Full details of conditions of acceptance and prices are given in the April and May issues of *Teintex* (21, 351-352 and 441 (1956)), and further information may be obtained from the Association pour la Détermination de la Solidité des Teintures et Impressions sur Textiles, 12 rue d'Anjou, Paris VIII, France.

## OBITUARY NOTICE

## Hubert Jennison

The Society suffered a great loss by the death on 28th March 1956 of its Honorary Treasurer, Hubert Jennison, M.C., A.M.C.T., A.R.I.C., F.S.D.C. It is with sorrow that I write these words about a good true friend, a great worker for the Society.

The Society honoured him by its confidence in electing him its Honorary Treasurer for more than twenty years (1933-1956) and bestowed on him in 1948 its highest honour, that of Honorary Membership. The Society has been fortunate in having a number of members who have given it many years of honorary service, but, in spite of his important activities in industry, none has given more time and interest to the Society.

Hubert Jennison was born in Manchester in 1892 and was educated at the Manchester Grammar School. He studied dyeing at the Manchester School of Technology under Dr. E. Knecht and Mr. J. Hübner, being awarded the Diploma of Associate-ship of the School. He was appointed Assistant Chemist in the laboratory of Thornton, Hannam, & Marshall Ltd., of Brighouse, a branch of the Bradford Dyers' Association Ltd., in 1911.

On the outbreak of war in 1914 he joined the West Yorkshire Regiment as a private, and rose from the ranks to become Captain of a trench mortar battery. He was twice wounded, was awarded the Military Cross, and was mentioned in despatches. He fought with the 29th Division at the Dardanelles, serving his country with honour and distinction.

At the end of the war he returned to the B.D.A. as Chemist at their Ripley West works in Bowling, Bradford. It was at this time that he was elected a member of the Society of Dyers and Colourists. His interest grew, and he was elected a member of the West Riding Section Committee in 1930; a year later he became Honorary Secretary of the Society, which office he held for two years, after which he was elected Honorary Treasurer and Convener of the Finance Committee. From 1948 he also served as Chairman of the Finance Committee. He was a member of a number of technical committees during this period; he was Chairman of the Education Committee from 1947 to 1949, and Convener of the Society's Medals Committee during 1947-1952 and 1955-1956.



No imagination is needed to realise the enthusiasm and the vast amount of time that "Jennie" gave to the interests of the Society's members. The Society gave thanks year by year to him over the many years when he was re-elected Honorary Treasurer at the Annual General Meeting. He certainly deserved such appreciation for his services to the Society for nearly a quarter of a century.

Hubert Jennison's business career was considerable and various. He remained at the Ripley West branch of the B.D.A. from 1919 until 1924, when he was transferred to B.D.A. Head Office laboratory. After a year there he returned to Ripley West as Works Manager; he was appointed Branch Director in 1933, which position he held until this works closed as a wool-dyeing and -finishing branch in 1943. From this date until 1948 he was Welfare Officer at Head Office (Labour Department), and from 1948 to 1953, when he retired, he was Branch Director of Shaw & Co. (Dyers) Ltd., of Greetland, another branch of the B.D.A.

In addition to his other duties, he was appointed Chairman of Works Council Conferences in January 1936, and he held this office for 15 years. He was also Chairman of the B.D.A. Inter-branch Games League from 1943 to 1952.

The present chairman of the B.D.A. Works Council Conference said at a recent conference: "Mr. Jennison held this office for a period of 15 years, during which he devoted much time and energy to every aspect of the Works Council movement, notwithstanding his many activities in other directions. His knowledge, tact, and sympathetic understanding of human needs won the confidence of these conferences and endeared him to all who had the privilege of knowing him. Mr. Jennison was a pioneer in all matters connected with welfare, in which he held a most sincere belief."

He was a man of many interests, beloved by those with whom he worked. He put great energy and thought into his many activities, for they were also his interests and pleasures. The Society has lost a true friend and a willing worker, but his name and influence will live on.

H. H. BOWEN

## New Books and Publications

## Basic Chemistry of Textile Colouring and Finishing

By S. R. Cockett and K. A. Hilton. Pp. vii + 191. London: National Trade Press Ltd. 1955. Price, 21s. 0d.

This is the companion volume to *Basic Chemistry of Textile Preparation* (cf. J.S.D.C., 71, 614 (Oct.

1955)), and assumes a knowledge of the fundamental chemistry and physics of textile materials outlined in the earlier volume. The scope of the present book is ambitious. There are chapters on dye chemistry, dye application and dyeing machinery, printing and the colouring of sundry non-textile materials including furs, paper, plastics,

food and cosmetics, and paints and inks. Finishing is dealt with in two long chapters, subdivided into finishes which are based primarily on the physical and chemical properties of the fibres present and finishes based on the application of resins, rubber, and similar compounds of high molecular weight. A comprehensive account of the present position of the theory of dyeing is included, and there is a chapter on testing and defects which includes an elementary scheme for identifying the dyes present on cellulosic fibres, wool, and silk.

The immediate impression gained is that the authors have been determined to include something about everything, and thus it is not surprising to find references to continuous dyeing methods, the Thermosol process, the cuprous ion method for dyeing polyacrylonitrile fibres, and a number of other recent developments. In this respect the present book is inevitably much more up to date than Whittaker and Wilcock's *Dyeing with Coal Tar Dyestuffs*, which from this standpoint is now incomplete. The text is plentifully supplied with formulae, graphs, and diagrams, particularly of machinery, which can be more useful than photographs if not oversimplified.

Any attempt to compress a great deal of fundamental information into a small compass and so cover a wide range of topics requires much effort and attention to detail, particularly if the book is to be of maximum value to students. In the present case there are rather too many instances of what appears to be hasty writing and lack of thorough revision. Thus in Table I on p. 2, in which the use of each class of dye is related to each class of fibre, the application of basic dyes to polyacrylonitrile fibres and of metal-complex dyes to regenerated protein fibres is omitted. The section dealing with the application of chrome dyes is particularly muddled. Thus, in connection with the chroming of wool, we read: "When wool is boiled in solutions of chromic acid or dichromates it reduces them to chromic oxide . . . The chromic oxide ( $\text{CrO}_3$ ) is itself further reduced to the sesquioxide or hydroxide ( $\text{Cr}_2\text{O}_3$  or  $\text{Cr}(\text{OH})_3$ )." Again, dealing with chrome dyes on silk: "As it [silk] has no cystine linkages and no reducing properties the lake formed contains chromium but not in the tervalent state suitable for the formation of the fibre-dye-chromium complex". Nylon is stated to "show little absorption of dichromate ions". "Carriers" are said to be of assistance when dyeing polyacrylonitrile fibres with acid dyes. Nylotan M is stated to be suitable as a blocking agent to be added to disperse dyebaths for nylon reserves in the dyeing of nylon-acetate rayon mixtures. In the section on textile printing it is stated that wool cloths are chlorinated "to increase the affinity of the wool" for acid dyes; and the "chloride of ammonium vanadate" is given as an oxygen carrier in aniline black printing, when presumably vanadium chloride or ammonium vanadate is meant. In Table III "azoic dye" is written instead of "acid dye". Among the books

quoted as sources are *Dyeing with Coal Tar Dyestuffs* and *The Theory and Practice of Wool Dyeing*; but it is the first (1946) edition of the latter instead of the second (1951), and the fourth (1942) and not the fifth (1950) edition of the former, which are listed. There are a number of other confusions and inaccuracies, all of which could have been eliminated if more time and care had been given to proofreading.

It should not, however, be thought that the value of the book is very seriously reduced by these defects. Anyone attempting the task of writing a textbook of this sort is performing a service of sufficient value to make up for quite a few minor faults. Since the book and its companion are completely complementary, it seems a pity that it was not possible to issue a single-volume first edition. The student must refer repeatedly to the first volume to comprehend much that is given in the second, and neither is so large that a single volume would be unwieldy. When a reprint or a second edition is called for, it would be worthwhile considering presentation within one cover. If the saving on binding were to make the complete work a little cheaper, those for whom it was primarily intended would surely not complain.

C. B. STEVENS

**Food Standards Committee  
Supplementary Report on Colouring Matters  
Recommendations relating to the Use of  
Colouring Matters in Food**

Ministry of Agriculture, Fisheries, and Food.  
Pp. 15. London: H.M.S.O. 1955. Price, 9d.

In 1954 the Food Standards Committee of the Ministry of Food issued a report prepared by its Preservatives Subcommittee and accepted its recommendation that certain specified colouring matters only should be permitted for use in food (cf. J.S.D.C., 71, 247 (1955)). Subsequently, representations were received that certain of the recommendations contained in the report should be reversed or modified. The present supplementary report discusses these representations and presents the Committee's conclusions.

In all main respects the supplementary report confirms the Committee's previous recommendations, reasoned argument being provided for its adherence to its views. Minor modifications are made in the recommendations in order to facilitate their application in practice, and in particular the recommended list of "Permitted Colours" has been revised, there being both additions and deletions.

The revised recommended list of "Permitted Colours" is presented as an appendix, in which the Subcommittee's reference number, the common name of the colouring matter, its scientific name, and its *Colour Index* (1924) number, if any, are given for each dye. The designation in terms of a hue and a number which was employed in the 1954 report has been dropped in favour of a number only and a common name.

M. LAPWORTH

## Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index

### I—PLANT; MACHINERY; BUILDINGS

#### Atmospheric Conditions in Viscose Spinning

A. Wilke

*Faserforsch. und Textiltech.*,  
7, 99-104 (March 1956)

Spinning rayon semi-continuously results in a more unfavourable atmosphere than when open bobbin spinning machines are used. Measures to improve atmospheric conditions are discussed. W.R.M.

#### PATENTS

#### Furnace Black Plant

Phillips Petroleum Co.

BP 748,585

Plant in which the carbon black dust is kept away from the terminals in the electro-precipitators used for separating the pigment from the effluent gases. C.O.C.

#### Mixers for Compounding Rubber, Plastics, Paint, Pigments, and like Materials

Francis Shaw & Co.

BP 748,197

The material to be compounded passes between two co-operating rotors. One rotor has two double helical teeth and the other three double helical teeth, the teeth being of any desired shape. C.O.C.

#### Handling and Storing Rolls of Material

Geo. H. Gascoigne Co.

BP 747,605

Apparatus which corrects skew more effectively and more accurately than prior mechanisms and does it with no appreciable lateral displacement or lateral travel of the cloth out of its correct path. C.O.C.

#### Molten-metal Drying Machine

Milwott Bleaching Co.

BP 747,822

The material passes through molten metal and then through an inert gas which extends over the exit from the molten metal. Preferably the material travels a considerable length horizontally through the metal bath to enable bubbles or gas trapped in the material to rise to the surface of the molten metal. C.O.C.

#### Stretching Webs Simultaneously Laterally and Longitudinally

DuP

BP 744,768

BP 746,386

Apparatus for stretching webs, particularly of thermoplastic materials, which enables the amount of lateral and longitudinal stretch imparted to be regulated to close tolerances. C.O.C.

#### Stenter

Sir James Farmer Norton & Co.

BP 747,106

Higher efficiency is obtained by directing the drying air through spaced streamlined nozzles extending transversely to the direction in which the cloth is passed and at such an angle to that direction that the air is projected partly on to the surfaces of the cloth and partly along the length of the cloth. The space between the mouths of the nozzles is closed or covered so that eddies cannot form between the nozzles in the air streams leaving the nozzles. This reduces the air pressure in the distribution trunking and therefore the power necessary to propel the air on to the cloth. C.O.C.

#### Electrical Drying Apparatus

Metropolitan-Vickers Electrical Co.

BP 745,700

Coatings etc. whose volatile constituents have a low flash-point, are subjected to infrared radiant heat with simultaneous projection of jets of air near to the heating elements so as to prevent vapour or gas gaining access to the elements which themselves are screened from the jets of air. C.O.C.

#### Self-balancing Laundry and Dry-cleaning Machinery

Manlove, Alliott & Co.

BP 748,077

#### Garment Press

J. C. Binns and S. Newbery

BP 748,548

#### Apparatus for Decorating or Printing on Non-planar Surfaces

J. Valiela

BP 746,100

The design is printed on a rubber sheet, from which it is transferred to the surface to be printed by placing it in contact with it under fluid pressure. C.O.C.

#### Silk Screen Printing Machine

L. G. Dubuit

BP 748,362

A machine for printing on convex, concave or other non-planar surfaces including non-rigid surfaces. C.O.C.

#### Photogravure Printing Plates or Rollers (IX p. 365)

### II—WATER AND EFFLUENTS

#### PATENT

#### Destroying Organic Components in Effluents

W. Busching

BP 748,527

The effluent is sprayed down a combustion tower so that the liquor is evaporated and the organic matter is burnt. The heat of this burning helps to evaporate the liquid. Some of the air at the bottom of the tower is withdrawn and returned to the upper part of the tower. This enables the effluent to be purified with small expenditure of heat and recovery of the inorganic constituents in it. C.O.C.

### III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

#### Table of Textile Auxiliary Products

St. Jost

*Textil-Rund.*, 11, 22-29 (Jan.),  
85-93 (Feb. 1956)

#### Heat of Micelle Formation with Sodium Dodecyl Sulphate

E. D. Goddard and G. C. Benson

*Trans. Faraday Soc.*,  
52, 409-413 (March 1956)

By means of an adiabatic calorimeter, measurements have been made of the heat of dilution of an aqueous solution of sodium dodecyl sulphate at 25°C. An abrupt change in the heat of dilution-concentration curve, at the critical micelle concentration, is not readily explicable in terms of current theories of micelle formation. W.R.M.

#### Alkyl Sulphates. I—Critical Micelle Concentrations of the Sodium Salts

H. C. Evans

*J.C.S.*, 579-586 (March 1956)

Critical micelle concn. of a number of Na alkyl sulphates, in which the total hydrocarbon chain contains 8-19 C atoms and the position of the sulphate group ranges from terminal to medial, are determined by repeated measurement of conductivity as the soln. is diluted. Data are also given for Na 1-tetradecylpentadecyl sulphate. The slopes of the plots are used in estimating the number of gegenions in the micelles. H.H.H.

#### Metal Derivatives of NN'-Diarylamidines

W. Bradley and I. Wright

*J.C.S.*, 640-648 (March 1956)

The ability of certain NN'-diarylamidines and related benzamidines, and of the dianil and the di-(p-chloroanil) of glutaraldehyde, 2-p-nitroanilinoquinoline, 2:4:6-triarylamino-triazines, the dianils of dimedone and indane-1:3-dione, benzimidazole, and di-(2-methyl-3-indenyl)methine, to form metal deriv. is investigated. Cuprous, cupric, silver, and mercuric deriv. are described and their structures discussed. H.H.H.

#### Organic Complex-forming Agents for Metals. I—Preparation of 4-Hydroxy- and 4:7-Dihydroxy-benzimidazoles and Related Compounds

E. S. Lane and C. Williams

*J.C.S.*, 569-573 (March 1956)

## PATENTS

**Textile Lubricant and Conditioning Agent**

Celanese Corp. of America BP 747,499  
A mineral oil is blended with a tertiary amine, an alkyl-phenol, an alkylolamine ester and/or amide of a fatty acid containing a straight chain of  $> 7$  C, and a partial phosphoric ester derived from a mixture of fatty alcohols each having  $> 7$  C. Such a composition has better antistatic properties than if a partial phosphoric ester derived from a single long-chain fatty alcohol is used. C.O.C.

**Salts of Hardenable, Basic, Tertiary Condensates—Finishing and Auxiliary Agents**

Ciba BP 748,016  
Water-soluble products are obtained by reacting together simultaneously or in any order (a) methylol compounds of aminotriazines or urea or their ethers with low-molecular alcohols, (b) non-basic aliphatic compounds containing a chain of  $> 6$  C and a reactive H atom attached to O, S or a non-basic N atom, (c) a primary or secondary amine or a tertiary amine containing a reactive H atom attached to O, S or a non-basic N atom. Their salts, including quaternary ammonium salts, have a wide range of uses as auxiliary and finishing agents, e.g. for imparting hydrophobic or crease- or shrink-resisting finishes to both cellulosic fibres and wool, and as softening agents. C.O.C.

**Ureido Derivatives of Vinyl Ethers and their Polymers—Finishing Agents**

Rohm & Haas Co. BP 747,184  
Compounds of formula  $\text{CH}_2=\text{CH}-\text{O}-\text{A}-\text{NR}-\text{CONH}_2$  (A = hydrocarbon of  $> 1$  C or is a chain between the O and N atoms containing O, S or N and interposes  $> 1$  C between each hetero atom; R = H, phenyl, or hydrocarbon which may contain ether-linked O) react with cellulose, polyvinyl alcohols and other polyhydroxy compounds to yield urea-modified materials which can be further reacted with formaldehyde. Thus those containing long-chain alkyl groups can be used for imparting water-repellency. C.O.C.

**Tanning Agents**

FBy BP 747,675  
Improved stable tanning agents are obtained by adding an aliphatic aldehyde and an *o*-chlorophenol (having at least one free *o*- or *p*-position) simultaneously but separately into a mono- or disulphonic acid or an aromatic hydrocarbon containing at least two condensed nuclei, while condensing the resulting mixture. C.O.C.

Tanning Agents and Tanning (XII p. 366)  
Dicyandiamide-Formaldehyde Resins for treating Leather (XII p. 366)

**IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS**

Sir William Henry Perkin—Birth of the Synthetic Dye Industry  
J. Simonsen

Biographical.

Research, 9, 166–169 (May 1956)  
C.O.C.

**Synthesis of *p*-isoCyanobenzonic Acid and *p*-iso-Cyanobenzenesulphonamide**

D. Samuel, B. Weinraub, and D. Ginsburg  
J. Org. Chem., 21, 376–377 (March 1956)

**Preparation of *oo*-Dihydroxyazo Compounds**

D. C. Freeman and C. E. White  
J. Org. Chem., 21, 379 (March 1956)

*o*-Aminophenol, and its 4- or 6-methyl deriv., are converted into the corresponding symmetrical azo dyes by diazotisation followed by rapid addition to a cuprammonium soln. reduced before use with hydroxylamine. The copper complexes formed are decomposed with hot conc. HCl or  $\text{H}_2\text{S}$ . H.E.N.

**Metal-complex Dyes. I—Manner of Action of Sulphamoyl and Methylsulphonyl Groups: Hammett's  $\sigma$ -Values and Solvation Effects**  
H. Zollinger and C. Wittwer

Helv. Chim. Acta, 39, 347–356 (March 1956)

From the acidity constants of benzoic acids (I) and anilinium ions (II) substituted in *m*- or *p*-position by  $-\text{SO}_2\text{CH}_3$ ,  $-\text{SO}_2\text{NH}_2$ ,  $-\text{SO}_3^-$ , or  $-\text{CN}$ ,  $\sigma$ -values are calc.

$\sigma$ -Values for II are greater than for I. The substituents therefore have a  $-M$  effect apart from their  $-I$  effect. Judged by these  $\sigma$ -values, the electric effects of  $-\text{CN}$  are very similar in magnitude to those of  $-\text{SO}_2\text{CH}_3$  and  $-\text{SO}_2\text{NH}_2$ , but, in general, these groups cannot be replaced successfully by  $-\text{CN}$  in dyes. The cause is demonstrated to be the difference in solvation by water, by means of an examination of the solubility, volatility in steam, and m.p. depression by moisture of the *p*-tolyl epd. H.E.N.

**Effect of Hydrogen Bonds on the Rate of Decomposition of Azo Dyes**

R. Mecke and D. Schmähl  
Naturwissenschaften, 42, 153–154 (1955);  
Chem. Abs., 50, 5293 (10 April 1956)

Hydroxyl *ortho* to an azo bridge forms a 6-membered chelate ring. The infrared spectrum of Methyl Red shows that in addition it has a hydrogen bond, between the *o*-COOH group and the  $\alpha$ -N atom of the azo group. Reduction with 6 mg. % aq. baker's yeast at pH 7 and 35°C. gave half-decomposition periods (hr.) of 4-dimethylaminoazobenzene of 20, 2-hydroxy-4-dimethylaminoazobenzene  $> 100$ , 4-hydroxyazobenzene 20, 2,4-dihydroxyazobenzene 70, 2'-carboxy-4-dimethylaminoazobenzene 0.5, 4'-carboxy-4-dimethylaminoazobenzene 3.5. Thus including the azo bridge in the chelate ring stabilises the azo bond, but ring closure to the  $\alpha$ -N atom of the azo bridge tends to make it more labile. C.O.C.

**Kinetics of the Reduction of some Azo Compounds**

N. R. Large and C. Hinshelwood.

J.C.S., 620–627 (March 1956)  
Titanous chloride reduction of substituted azobenzenes in aq. alcohol is found to be of the first order with respect to the azo epd. and to titanous chloride, and the rate varies in a complex way with the HCl concn. In some examples the two stages of reduction, azo  $\rightarrow$  hydrazo, and hydrazo  $\rightarrow$  amines, are comparable in speed, and in the case of 2:2':4-trihydroxyazobenzene the kinetics of the consecutive reactions is worked out. The influence of substituents is complex, and appears to be the result of opposing effects, viz. the process of electron transfer from the reducing agent, and the equilibrium between protonated and non-protonated azo species. There are indications that changes in the former are reflected mainly in the activation energy, and in the latter in the non-exponential factor in the Arrhenius equation. This results in the absence of additivity in free energies of activation. The reduction of azobenzene by stannous chloride is included for comparison. H.H.H.

***o*-Mercapto-azo Compounds**

A. Burawoy and A. Chaudhuri  
X—Reactions of Azobenzene-2-sulphenyl Bromide and its Derivatives with Malonic Acid, Acetone, and Acetophenone

J.C.S., 648–652 (March 1956)  
At room temp. azobenzene-2-sulphenyl bromide and its 2-nitro, 4-nitro, and 2-benzylthio deriv. condense slowly with malonic acid, acetone, or acetophenone in aq. or ethanolic soln. to yield the corresponding (2-aryl-azophenylthio)-acetic acids, acetonyl 2-arylazophenyl sulphides, and 2-arylazophenyl phenacyl sulphides respectively, the condensation with malonic acid being accompanied by partial decarboxylation. The tendency of these products to decompose in presence of acid to the corresponding arylamines and benzothiazole-2-carboxylic acid, 2-benzothiazolyl methyl ketone, and 2-benzothiazolyl phenyl ketone respectively offers a novel route for the preparation of benzothiazole deriv.

**XI—Action of Alkali on Azobenzene-2-sulphenyl Bromide and its Derivatives**

Ibid., 653–658  
According to conditions, azobenzene-2-sulphenyl bromide and its deriv. react with aq.  $\text{NaHCO}_3$ , aq.  $\text{NaOH}$ , and (wet) liquid  $\text{NH}_3$  to give the corresponding disulphides and either disulphoxides or sulphinates. The reactions of 2-phenylazonaphthalene-1-sulphenyl bromide with alkalis are complicated by the tendency of di-(2-phenylazo-1-naphthyl) disulphoxide to decompose readily into di-(2-phenylazo-1-naphthyl) sulphide and sulphur dioxide. H.H.H.

### Aromatic Azo Compounds. VIII—Intramolecular Hydrogen Bonding in 8-Hydroxyquinoline

G. M. Badger and R. G. Buttery

*J.C.S.*, 614-616 (March 1956)

8-Hydroxy-5-phenylazoquinoline (I) is shown to exist in soln. as an equilibrium mixture of the azo and phenylhydrazones tautomers with the former predominant. With 8-hydroxy-5-phenylazoquinoline *N*-oxide (II) little or none of the phenylhydrazone tautomer is present. It is suggested that the position of the equilibrium is affected by the presence of a weak intramolecular hydrogen bond in I and a very strong bond of the same type in II.

H.H.H.

### Theory of Colour—Seto Blue VE, a Sterically Hindered 2:2':2''-Trimethyl-Crystal Violet

W. Theilacker and W. Berger

*Chem. Ber.*, 89, 965-970 (April 1956)

A structure for triphenylmethane dyes in which two phenyl rings are, as far as is possible, coplanar, and in which the third ring (I) is displaced out of the plane to the extent demanded by crowding, is suggested as an alternative to the propeller structure. *o*-Substituents cause I to leave the plane still further, and this explains their effect on the colour and on the fastness to alkali. The absorption spectrum of Seto Blue VE ( $\lambda_{\max} = 606 \text{ m}\mu$ ) is recorded and lies between those of Crystal Violet (584  $\text{m}\mu$ ) and Malachite Green (625  $\text{m}\mu$ ), with greatly reduced intensity.

H.E.N.

### Theory of Colour—Triarylmethane Dyes of the Diphenyl Series—II

W. Theilacker, W. Berger, and P. Popper

*Chem. Ber.*, 89, 970-983 (April 1956)

A Friedel-Crafts reaction between 4'-nitrodiphenyl-4-carboxyl chloride and diphenyl, followed by nitration, yields 4:4'-bis-*p*-nitrophenylbenzophenone. This is reduced, methylated, and condensed with 4'-dimethylaminodiphenyl-4-magnesium iodide to form the carbinol (I) of the diphenyl analogue of Crystal Violet. I dissolves in sulphuric or perchloric acid with a deep red colour, destroyed on dilution. A very dil. soln. in acetic acid-anhydride is almost colourless, but becomes intensely blue to blue-green at the b.p., fading again on cooling. This behaviour is very similar to that of the carbinol (II) of the diphenyl analogue of Malachite Green (Theilacker and Schmid, cf. *J.S.D.C.*, 67, 460 (1951)), a new synthesis of which is described. The following  $\lambda_{\max}$  in acetic acid-anhydride (4:1; at different temp.) are recorded: Malachite Green 625, II  $\sim 700$ , Crystal Violet 584, I 643, diphenyl-4'-dimethylaminodiphenyl-4-carbinol  $\sim 850$ , and 4-dimethylaminodiphenylcarbinol 457  $\text{m}\mu$ . In discussing the results, the lower stability of trisdiphenylmethane dyes is attributed to the participation of quinonoid structures, which are less stable than in the case of phenyl rings.

H.E.N.

### Diphenyl-4'-amino-4-diphenylcarbinol

W. Theilacker and H. M. von Blumenroon

*Chem. Ber.*, 89, 984-986 (April 1956)

4-Aminotriphenylcarbinol is diazotised and coupled with nitrobenzene in aq. acetic acid containing sodium acetate, yielding 2-4% diphenyl-(4'-nitrodiphenyl-4)-carbinol (I). Hodgson and Marsden's method (*J.C.S.*, 208 (1940)) increased the yield to 7-15%. Reduction gave the desired product, solutions of which in sulphuric and 70% perchloric acids are orange-red, in formic acid orange-yellow, in acetic acid practically colourless, becoming reversibly green on heating.

H.E.N.

### Monodiphenyldiphenylmethane Dye

F. Klages and H.-J. Mandler

*Chem. Ber.*, 89, 987-995 (April 1956)

Friedel-Crafts condensation of 4-dimethylaminodiphenyl and benzoyl chloride, followed by addition of *p*-dimethylaminophenyllithium, gives the carbinol of the phenyl homologues of Malachite Green. Acid forms with it an intense red colour corresponding to the yellow "acid salt" of Malachite Green. The true dye, which was black-blue, can be obtained only in 40% alcohol. From absorption spectra, it is shown to be present to a maximal extent ( $\sim 1\%$ ) in aq. soln. at pH 3.5. Its  $\lambda_{\max}$  lies at 625  $\text{m}\mu$ . An attempt is made to interpret the data of this and the previous papers by Theilacker *et al.* H.E.N.

### Condensation of Acridone with Tertiary Aromatic Amines and the Ultraviolet Absorption Spectra of the Products

R. M. Acheson and M. J. T. Robinson

*J.C.S.*, 484-489 (March 1956)

From the ultraviolet absorption spectra of the condensation products of acridone with dimethylaniline, triphenylamine, *NN*-dibenzylaniline, *m*-bromodimethylaniline, dimethyl- $\alpha$ - and - $\beta$ -naphthylamine, and *NN*-dimethyl-*o*-toluidine, it is suggested tentatively that the colours of 5-*p*-aminoacridinium ions are due to intramolecular charge-transfer phenomena.

H.H.H.

### Synthesis of 1-Substituted Phenothiazines by Thionation of Diarylamines

S. P. Massie and P. K. Kadaba

*J. Org. Chem.*, 21, 347-348 (March 1956)

### Catalytic Syntheses of Heterocycles. VIII—Dehydrocyclisation of *o*-Ethylbenzenethiols to Thianaphthens

C. Hansch, B. Schmidhalter, F. Reiter, and W. Saltonstall

*J. Org. Chem.*, 21, 265-270 (March 1956)

### Improving the Quality of Sulphur Dyes as a Result of Study of their Physicochemical and Colloidal Properties

E. M. Aleksandrova and V. K. Laryushkina

*Kolloid. Zhur.*, 17, 409-414 (1955);

*Chem. Abs.*, 50, 4509 (25 March 1956)

The poor fastness to wet rubbing, instability towards electrolytes and presence of large insoluble residues in sulphur dyes are caused by introduction of NaCl during their manufacture causing them to be coarsely dispersed. If the  $\text{Na}_2\text{S}_2\text{O}_3$  originally present in the mix is not displaced by NaCl or if a wetting or dispersing agent is added at an early stage of production, the resulting dyé is more stable.

C.O.C.

### Synthesis of 4-Methoxy- and 4-Hydroxy-1-acylaminoanthraquinone Derivatives

T. Hayashi and M. Shitama

*J. Chem. Soc. Japan, Ind. Chem. Sect.*,

58, 116-117 (1955);

*Chem. Abs.*, 50, 4513 (25 March 1956)

4-Methoxy-1-aminoanthraquinone (1 mole) and an acyl chloride (2 moles) (acyl = benzoyl, *p*- $\text{RC}_6\text{H}_4\text{CO}$  ( $\text{R} = \text{CH}_3$ ,  $\text{CO}$ ,  $\text{Cl}$  or  $\text{NO}_2$ ) or *m*- $\text{O}_2\text{NC}_6\text{H}_4\text{CO}$ ) were heated in nitrobenzene or *o*-dichlorobenzene at  $140^\circ\text{C}$ . for 1 hr. in presence of pyridine (1.1 mole). A  $\text{p-C}_6\text{H}_4\text{C}_6\text{H}_4\text{CO}$  derivative was obtained similarly from the chloride prepared from 1.5 moles of the acid and 2.1 moles of  $\text{SOCl}_2$ . Similarly, 4-hydroxy-1-aminoanthraquinone was treated at  $140^\circ\text{C}$ . for 2 hr. with each of the above acyl chlorides in the *o*-dichlorobenzene. All products (purified from *o*-dichlorobenzene) were orange to red direct dyes. The yields of crude dye were 90-99%. Their fastness to light and washing are given.

C.O.C.

### Anthraquinonoid Dyes. V—3-Arylaminoalizarins

K. Murata, K. Harada, and K. Takiyama

*Bull. Fac. Eng., Hiroshima Univ.*,

4, 387-390 (1955);

*Chem. Abs.*, 50, 5293 (10 April 1956)

Na acetate (0.2 g.) and  $\text{CuCl}_2$  (0.07) were added to 3-aminoalizarin (0.5) and bromobenzene (1) in nitrobenzene (15), refluxed for 20 hr., the solvent distilled off and the residue filtered, treated with dil. HCl and then with dil. alkali to give a 28% yield of 3-anilinoalizarin, which vat-dyed rayon yellow-orange and wool dark purple red with a Cr mordant, yellow-orange with Sn and red-purple with Al. Similarly were prepared 3-(4-nitroanilino)alizarin (22% yield) a greyish yellow vat dye for rayon and brownish violet, yellowish orange and greyish violet mordant dye with Cr, Sn and Al respectively; 3-(2,4-dinitroanilino)alizarin (68%) pale brown vat dye, orange yellow, pale orange yellow or pale orange yellow mordant dye; 3-(4-carboxyanilino)alizarin (29%) pale brown vat dye, greyish violet, pale orange yellow and pale pink mordant dye; 3-(*p*-sulphanilino)alizarin, black, yellowish orange and blackish purple mordant dye; 3-(1-naphthylamino) (39%) alizarin pale yellowish orange vat dye, pale violet brown, pale yellowish orange and dark red mordant dye; 3-(2-naphthylamino)alizarin (32%) pale yellowish orange vat dye, pale violet brown, pale yellowish orange and pale pink mordant dye; 3-(1-anthraquinonylamino)alizarin (73%) greyish brown vat dye, brown violet, grey

and greyish purple mordant dye; 3-(2-anthraquinonylamino)alizarin (30%) brown vat dye, brown, greyish orange and brown mordant dye; 3-(8-amino-1-anthraquinonylamino)alizarin (67%) brown vat dye, pale orange, —, and pale brown mordant dye; 3-(2-methyl-1-anthraquinonylamino)alizarin (22%) pale pink vat dye, reddish purple, pale orange brown and — mordant dye; 1:8-bis(3-alizarylamino)anthraquinone (19%) brown vat dye, dark reddish purple, pale orange and pink mordant dye; 1:5-bis(3-alizarylamino)anthraquinone (57%) brown vat dye, dark purple, pale orange and pink mordant dye; 2:6-bis(3-alizarylamino)anthraquinone (68%) greyish brown vat dye, greyish brown violet, pale yellowish orange and reddish orange mordant dye; 1:5-bis(3-alizarylamino)-4:8-dihydroxyanthraquinone (35%) greyish violet vat dye, greyish violet, greyish brown and pale brown mordant dye; di-3-alizarylamine (35%) greyish brown vat dye, greyish black, pale orange and brown mordant dye.

C.O.C.

### Absorption Spectra of Indigoid Dyes

P. W. Sadler

*J. Org. Chem.*, 21, 316-318 (March 1956)

Substituents in either the 5- or 6-positions produce equal but opposite effects in the longest wavelength of maximum absorption. The effects of substituents in the 4- or 7-positions are found to be unpredictable. Spectral characteristics for several new deriv. are reported.

H.E.N.

### Metal Hydroxide Clathrate Compounds

W. Feitknecht and H. Bürki

#### I—Basic Zinc Salts of Naphthol Yellow S

*Helv. Chim. Acta*, 39, 564-575 (March 1956)

These are formed when soln. containing zinc salts and Naphthol Yellow S (I) are incompletely pptd. by NaOH or when zinc hydroxide (amorphous or  $\beta$ -form) is treated with the zinc salt of I. Their stability is intermediate between those of the basic chloride and the basic nitrate. Increasing the proportion of I increases the distance between the layers of zinc hydroxide in freshly prepared material from 14.2 to 24.2 Å. On ageing, the separation of the layers can fall to 12.8 Å.

#### II—Their Structure

*Ibid.*, 576-584

The mol. of zinc salt of I fill the gaps between the layers of zinc hydroxide and are probably oriented perpendicular to them.

#### III—Basic Salts of Naphthol Yellow S and Bivalent Metals

*Ibid.*, 584-589

The metals studied were Cd, Mn, Co, Ni, and Cu.

#### IV—Basic Zinc Salts of Other Aromatic Acids

*Ibid.*, 589-595

Cpd. investigated were *o*- and *p*-nitrophenol, picric, benzoic, salicylic, and benzenesulphonic acids, Erioflavine 3G, Erioglauine A, and Helvetia Blue. Layer structures were formed, the distance between the sheets of zinc hydroxide varying from 11.4 to 28 Å, depending on the organic acid.

H.E.N.

### Exchange Reactions and Magnetic Susceptibilities of Complex Salts. IV—Correlation with Absorption Spectra

H. C. Clark and A. L. Odell

*J.C.S.*, 520-524 (March 1956)

Syntheses in the Carotenoid Series. XXXI—Synthesis of 10:10'-*cis*-Bixin Methyl Ester; a Contribution to the Stereochemistry of the *cis-trans* Isomeric Bixins

H. H. Inhoffen and G. Raspé

*Annalen*, 592, 214-221 (1955)

### Carotenoid Syntheses

#### IV—Synthesis of *iso*Zeaxanthin

O. Isler, H. Lindlar, M. Montavon, R. Rüegg, and P. Zeller

*Helv. Chim. Acta*, 39, 449-454 (March 1956)

#### V—Application of Whiting's Reaction

O. Isler, M. Montavon, R. Rüegg, and P. Zeller

*Ibid.*, 454-462

Starting from various  $C_{40}$  diols, 7:7'-dihydro- $\beta$ -carotene and other carotenoids are synthesised. In addition, a new preparation of  $\beta$ -carotene from  $\beta$ - $C_{40}$  ketone, acetylene, and Vitamin A aldehyde is described.

### VI—Synthesis of Crocetin Dialdehyde, Lycopene, and $\beta$ -Carotene

O. Isler, H. Gutmann, H. Lindlar, M. Montavon, R. Rüegg, G. Ryser, and P. Zeller

*Ibid.*, 463-473

H.E.N.

### Comparison of Zinc Oxide Pigments produced by Different Methods

B. L. Davydovskaya and N. K. Mel'nikova

*Byull. Obmen. Opytom i Lakokrasochnoi Prom.*, (4), 12-17 (1953);*Referat. Zhur. Khim.*, 9800 (1953);*Chem. Abs.*, 50, 5304 (10 April 1956)

Zinc oxide prepared by oxidation of Zn vapour was compared with those obtained by treating  $ZnSO_4$  vapour with ammonia or by calcining the basic carbonate. The colour, covering power and intensity of colour differed very little with the exception of those obtained from the basic carbonate precipitated from an ammonia complex. Differences were seen in brilliancy, photochemical activity, lowest degree of dispersion and in connection with the last, the greatest atmospheric stability. Industrial samples of  $ZnO$  had insufficient atmospheric stability but this could be increased considerably by additional calcination of the samples at 900°C. Electron-microscopical studies show that the pigment obtained from the first mentioned method are needle-like crystals whereas the particles of the others are spherical.

C.O.C.

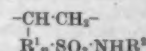
### PATENTS

#### Polymeric Colour Couplers

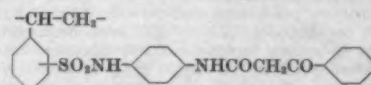
Kodak

BP 747,589

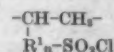
Colour couplers of formula—



( $R^1$  = group containing Ar;  $R^2$  = group containing Ar and a coupling function;  $n$  = 0 or 1), e.g.—



are obtained by treating a compound of formula—



with a colour coupler containing an amino group available for substitution. They yield dyes of high colour intensity for the weight of polymer used.

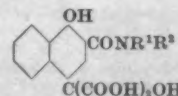
C.O.C.

#### Cyan Colour Formers

ICI

BP 747,628

Compounds of formula—



( $R^1$  = H or subst. or unsubst. hydrocarbon;  $R^2$  = subst. or unsubst. hydrocarbon), e.g. 1-hydroxy-2-N-cetylcarbamyl-naphthyl-4-tartronic acid, are colour formers which can be added to the colour developer or incorporated in the photographic emulsion.

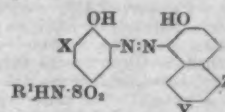
C.O.C.

Red, Violet, and Grey Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.

ICI

BP 748,421

The complexes containing 1 atom of Cr or Co to 2 mol. of monoazo compounds—



(X = H, Hal or  $NO_2$ ;  $R^1$  = H, Aryl of benzene series free from sulphonyl groups, Alk of < 4 C, or hydroxyalkyl of 2 or 3 C; of Y and Z, one = H, the other =  $NH \cdot CO \cdot R^2$ , where  $R^2$  = Alk or O-Alk of 1-4 C or  $O \cdot C_2H_4 \cdot OCH_3$  or

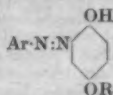
$O-C_2H_4-OC_2H_5$ ) dye wool, nylon etc. red, violet, or grey from neutral or weakly acid baths. Thus 2-aminophenol-4-sulphonamide is diazotised and coupled with an alkaline soln. of 1-carboethoxyamino-6-naphthol, and the monoazo compound so formed is boiled with cobaltous chloride in aq. ethylene glycol. The product dyes wool bordeaux and nylon dull red violet. E.S.

#### Azoic Dyes—Triazine from Tetrazotised Dianisidine and *N*-isopropylglycine

American Cyanamid Co. USP 2,712,005  
The triazine compound from tetrazotised *o*-dianisidine and 2 mol. of *N*-isopropylglycine is more stable than the *N*-allylglycine compound, and of higher solubility than the *N*-methyl- or *N*-ethyl-glycine compound. E.S.

#### Metallisable Monoazo Dyes from Quinol Monoethers

American Cyanamid Co. USP 2,709,697-8  
The metal complexes, prepared in substance or on the fibre, of monoazo compounds—



(Ar = a sulphonated aryl radical containing a group such as OH, COOH, etc., capable of entering into metal-complex formation, *ortho* to the azo link; R = Alk, Alkylene, aryl, or aralkyl) are chiefly blue, green, and black wool dyes. Thus diazotised 2-amino-4-nitrophenol-6-sulphonic acid is coupled with *p*-methoxyphenol in presence of aq. NaOH and pyridine. The monoazo compound so formed dyes wool dark green by the afterchrome process. Or it may be refluxed with a solution prepared by boiling chromic acid dissolved in aq. acetic acid with glucose to give the Cr complex, which dyes wool green. E.S.

#### Metallisable Azo Direct Dyes

ICI BP 747,872

Metallisable direct dyes similar to those of BP 282,107 (J.S.D.C., 45, 224 (1929)) prepared by sulphating *o*-hydroxyazo compounds with chlorosulphonic acid or its esters, are made by coupling diazotised sulphuric esters of *o*-aminophenols or *o*-aminonaphthols with suitable coupling components, e.g. arylides of 3-hydroxy-2-naphthoic acid. These diazotised sulphuric esters couple more rapidly than the aminophenols themselves. Disazo dyes may be made by using coupling components such as *NN'*-bis-acetoacetyl-*o*-toluidine, or by using e.g. J acid, which gives an aminomonoazo compound which can be then diazotised and coupled with a suitable end component. Thus *o*-nitrophenol is sulphated, and the nitro group then reduced to an amino group. The *o*-aminophenyl sulphate so formed is diazotised at  $-5^\circ\text{C}$ . and coupled with 3-hydroxy-2-naphtho- $\beta$ -naphthylamide to give the monoazo compound, which dyes cotton rubine by the aftercoppering method. E.S.

#### Blue Monoazo Disperse Dyes

Eastman Kodak Co. BP 735,897  
This corresponds to USP 2,683,708-9 (J.S.D.C., 71, 556 (1955)).

#### Blue Anthraquinone Disperse Dyes

Celanese Corp. of America USP 2,713,060  
Modification of USP 2,480,260 by treating the dye dissolved in an organic solvent with an alkaline agent, e.g. NaOH. This removes any solvent-insoluble material present in the dye and hydrolyses any other groups present. The products have better dyeing properties than the untreated dye. The treatment also improves the dyeing properties of any azoxy compound present.

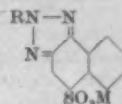
Thus 4:8-dinitroanthrarufin (20 parts by wt.), aniline (81.5) and water (3) are stirred for 4 hr. at  $105-110^\circ\text{C}$ ., cooled and poured into ethyl alcohol (160). 32% HCl (180) and water (150) are added. The dye, 4-anilino-8-nitroanthrarufin, is filtered off and washed with a mixture of equal volumes of HCl and water. The dye (20) is added to dioxan (60), water (120) and 50% aq. NaOH (30), refluxed for 12 hr. and filtered while hot. Water (400) is added to the filtrate, and glacial acetic acid (26) added to precipitate the dye. The product is a blue disperse dye. C.O.C.

#### Fluorescent Brightening Agents

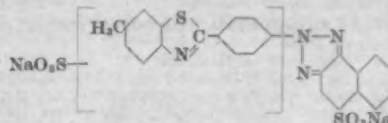
DuP

USP 2,713,054

Compounds of formula—



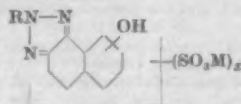
(R = 2-phenylbenzothiazole or a homologue or a monosulpho derivative of these; M = H, alkali metal or  $\text{NH}_4$ ) are prepared by diazotising 2-(*p*-aminophenyl)-benzothiazole, dehydro-thio-*p*-toluidine or dehydro-thio-*m*-xylidine or their monosulpho derivatives and coupling in acid medium with either 1-amino- or 2-aminonaphthalene-4-sulphonic acid. The resulting *ortho*-aminomonoazo compound is then oxidised to a triazole, e.g.—



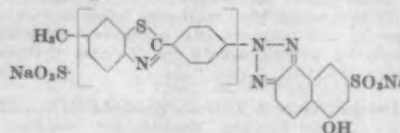
They have bluish fluorescence and are fast to chlorine.

USP 2,713,055

Compounds of formula—



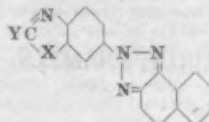
(x = 1 or 2), e.g.—



have bluish fluorescence and are fast to chlorine.

USP 2,713,056

Compounds of formula—



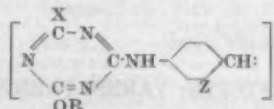
(X = O, S or imino; Y = a neutral substituent, e.g. Alk or Ar), e.g. 2-(2-methyl-6-benzothiazolyl)-2H-naphtho-(1:2)-triazole, have similar properties. Those having only sulphonyl substituents or no substituents are applied to nylon from aqueous dispersion. C.O.C.

#### Fluorescent Brightening Agents

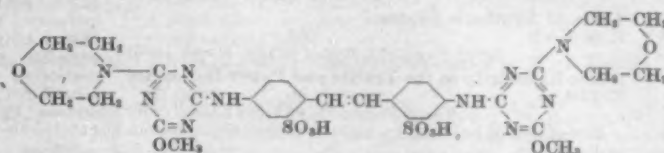
General Aniline

USP 2,713,046

Compounds of formula—



(R = Alk; X = OR or  $\text{NH}_2$ , residue of a primary or secondary amine, or a cyclic amine; Z = COOM or  $\text{SO}_3\text{M}$ ; M = H or cation), e.g.—



are substantive to cellulose and show a bluish fluorescence. They are specially suitable for use in soap and synthetic detergents.

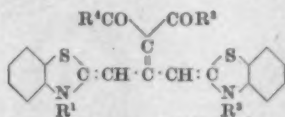
C.O.C.

### Cyanine Dyes

Ilford

Dyes of formula—

BP 747,083



( $\text{R}^1$ ,  $\text{R}^2$  and  $\text{R}^3$  = Alk;  $\text{R}^4$  = Alk or alkoxy;  $\text{R}^3 + \text{R}^4$  together have < 12 C; the benzene rings may contain one or two Hal, Alk, alkoxy or Ar groups) are photographic sensitizers having strong sensitivity in the green and red regions of the spectrum.

C.O.C.

### Powdered Calcium Carbonate coated with a Quaternary Salt or Base

Produits Chimiques et Minéraux

BP 747,842

Calcium carbonate is rendered much more suitable as a filler if it is coated with a compound of formula  $\text{XNR}^1\text{R}^2$ .  $\text{R}^1\text{R}^2$  (X = OH, or acid ion or radical;  $\text{R}^1$ ,  $\text{R}^2$ ,  $\text{R}^3$ ,  $\text{R}^4$  = same or different organic radical). Thus chalk (20 g.) of average particle size < 2  $\mu$  is made into a colloidal suspension with tetracyclpyridinium bromide (0.4 g.) and water (100 c.c.) To the suspension is added 25% aq. Na oleate (20 c.c.), and the precipitate is dried and milled.

C.O.C.

### Pigment Pastes

Hunter Metallic Products Corp.

USP 2,713,006

A metallic or non-metallic pigment is pasted with a liquid chlorinated wetting agent. The products are brighter and have greater specular gloss than hitherto known paste pigments. Metallic, e.g. Cu, bronze, and Fe, pigments so pasted lose no lustre even on prolonged exposure to air, moisture and corrosive environments. The preferred wetting agents are chlorinated diphenyls containing 64% Cl.

C.O.C.

### Metal Derivatives of NN'-Diarylamidines (III p. 357)

Organic Complex-forming Agents for Metals. I—

Preparation of 4-Hydroxy- and 4:7-Dihydroxy-

benzimidazoles and Related Compounds (III p. 357)

Pearl Essences (V below)

Yellow Schiff's Bases as Irreversibly Dischargeable

Photographic Filter and Antihalation Dyes (IX p. 365)

## V—PAINTS; ENAMELS; INKS

PATENT

### Pearl Essence

Mearl Corp.

USP 2,713,004

Guanine, lead chloride, mercurous chloride or other crystalline compound having a refractive index different from that of the medium in which it is to be dispersed is prepared in plate-like crystals by subliming it, preferably under high vacuum, and condensing the vapour on a sheet of large area. The sheet is preferably coated with the film-forming vehicle in which the pigment is to be dispersed. The pigment is stripped from the sheet and dispersed in the appropriate vehicle to yield pearl essence.

C.O.C.

Pigment Pastes (IV above)

## VI—FIBRES; YARNS; FABRICS

### Textile Fibres

P.-A. Koch

Textil-Rund., 11, 30-31 (Jan. 1956)

A classification of natural and man-made fibres.

S.R.C.

### Table of Synthetic Fibres

P.-A. Koch

Textil-Rund., 11, facing p. 130 (March 1956)

### Static Electricity in the Textile and Paper Industries

E. Bitterli

Textil-Rund., 11, 198-201 (April 1956)

Prevention of build-up by high-frequency air ionisation is illustrated.

S.R.C.

### Freezing Point Depression in Assemblages of Moist Fibres

J. M. Preston and G. P. Tawde

J. Textile Inst., 47, T154-T165 (March 1956)

Moist fibres are cooled, using solid  $\text{CO}_2$ , and studies made of cooling curves, rate-of-cooling curves, and the relation between regain and freezing point. Both natural and man-made fibres, including regenerated cellulose and protein fibres, are used. Evidence of freezing is obtained only when the moisture is above a limiting value, e.g. between 37.5 and 41% for viscose rayon. Results are discussed in terms of crystallinity, sites available for moisture adsorption, and effects of cross-linking.

J.W.B.

### Solubility and Swelling of Rayon Pulp in Caustic Soda Lyes

B. Gohlke

Faserforsch. und Textiltech., 7, 111-119 (March 1956)

Rayon pulp is treated with NaOH soln. of different concn. at temp. of 20-60°C. The resistant, soluble-precipitable, and soluble-non-precipitable fractions of the pulp are estimated after removal from the solution without rinsing with solution or water, and the values compared with those obtained after rinsing with water. Swelling values in NaOH soln. are obtained and compared with solubility data under corresponding conditions of concn. and temp.

W.R.M.

### Thermal Conductivity of Selected Flannel and Serge Fabrics before and after Wear

E. L. Phelps, M. A. Morris, and L. O. Lund

Amer. Dyestuff Rep., 45, 181-183 (26 March 1956)

### Crystallinity Studies of Polyamides

I. Sandeman and A. Keller

J. Polymer Sci., 19, 401-435 (March 1956)

Examination of the infrared absorption of 6,10-, 6,6-, and 6-nylons showed bands associated with crystalline order. A method is deduced for comparing the intensity of an infrared band in a drawn polymer with that for a disoriented sample. The crystallinity of 6,10-nylon is determined by infrared and specific volume measurements, and the effects of annealing and drawing are studied. Proportionality between changes in specific volume and infrared absorption holds up to an annealing temperature of 180°C. Cold drawing is found to be connected with a reduction in crystallinity. The infrared spectrum of 6-nylon is examined, and several bands are classified as crystalline or non-crystalline. The variation of these bands is studied when the polymer is annealed, drawn, and melted. X-Ray results suggest a new type of ordering intermediate between randomness and full crystalline order, and two infrared absorption bands are found to be associated with this state of order. Direct evidence is obtained for order in the quenched amorphous polymer. An absolute density of 1.052 g./c.c. is deduced for amorphous 6,10-nylon.

W.R.M.

### Acrylic Fibres

#### XI—Preparation of Solutions of Polyacrylonitrile for Spinning

A. Hunyar and H. Fark

Faserforsch. und Textiltech., 7, 131-132 (March 1956)

#### XII—Mixtures of Polyacrylonitrile and Cellulose Acetate

V. Grobe

Ibid., 132-133

Polyacrylonitrile and secondary cellulose acetate dissolve in dimethylformamide to give clear solutions the viscosity of which falls with increasing cellulose acetate content. The properties of fibres spun from such solutions containing 5-20% cellulose acetate are examined and compared with those of pure acrylonitrile fibres. Tenacity decreases only slightly with increasing cellulose acetate content. Extension before break remains approximately constant. Flexibility increases. The fibres can be dyed with various dyes and have a silky lustre. Examination of cross-sections shows complete penetration of dye. Extraction of fibres containing 20% cellulose acetate with acetone for 14 hr. caused 8.5% loss in weight by dissolution of cellulose acetate. Dyeing properties were reduced by the extraction, but textile properties were not greatly changed.

W.R.M.

## PATENTS

**Stabilising the Viscosity of Polyamides**

British Nylon Spinners

BP 747,614

Aromatic  $\alpha$ -diamines, e.g.  $\alpha$ -phenylenediamine, are useful as viscosity stabilisers in the manufacture of polyamides. They have the advantage that they also impart increased affinity for acid dyes. C.O.C.

**Improving the Dyeing Properties of Synthetic Polypeptides**

Courtaulds

BP 747,901

Synthetic polypeptides containing at least one anhydro-carboxy amino acid having a hydrophobic chain containing an ester group are treated with a base so as to convert the hydrophobic side-chain either partly or wholly into hydrophilic side-chains. C.O.C.

**Acrylonitrile-Glycerol  $\alpha$ -Alkenyl Ether Copolymers**

Chemstrand Corp.

BP 747,798

Copolymers yielding fibres of improved receptivity for disperse, vat, sulphur, acid, basic and direct dyes are obtained by copolymerising glycerol  $\alpha$ -alkenyl ethers containing only one ether group per molecule with acrylonitrile. Preferably the copolymer will contain 90-98% of acrylonitrile. C.O.C.

Variations in the Electrokinetic Potential of Acetate Rayon and Hydrophobic Substances in Solutions of Surface-active Compounds, with reference to Dyeing Theory (VIII p. 364)

**VII—DESIZING; SCOURING; CARBONISING; BLEACHING****Soil-resistant Treatment of Fabrics**J. M. Salisbury, T. F. Cooke, E. S. Pierce, and P. B. Roth  
*Amer. Dyestuff Rep.*, 45, P 190-P 202 (26 March 1956)

The nature of soil and the mechanism of soiling are briefly discussed, and photomicrographs are shown to emphasise the effects of fibre surface structure. A laboratory method for measuring dry soiling properties of fabric is described, and the results are compared with actual wearing trials on carpets made from different fibres. Trials with a new soil-resistant finish—Juvenon R (American Cyanamid Co.)—are described in detail, together with its mode of application. It probably acts as a soil barrier, its colourless particles occupying sites which would otherwise be filled with soil. J.W.B.

**Surface Properties of Carboxymethylcellulose**

H. Stupel

*J. Polymer Sci.*, 19, 459-462 (March 1956)

Carboxymethylcellulose (CMC) is an important builder for detergents and increases whiteness retention. Fluorescence microscopy may be used to determine adsorption of CMC. In laundering, CMC is not adsorbed by fibres but is attached to the soil. At a high concentration in neutral media, and in acid media, CMC is adsorbed by the fibre. W.R.M.

**Influence of Carboxymethylcellulose in Detergent Solutions on the Ash Contents of Textile Fabrics**

K. H. Tan and K. J. Nieuwenhuis

*Bull. Inst. Text. France*, (58), 69-88 (Feb. 1956)

Results of a large number of washing tests to assess the effect of carboxymethylcellulose (CMC) on the ash contents of laundered textile fabrics (cotton, linen, rayon) are summarised. Some of the data are original; others are taken from the literature. In almost all cases the addition of CMC to the washing preparations results in a significant decrease in the ash content of the laundered articles. The effect is most marked when hard water is employed, whether the washing agent be soap + alkali, synthetic detergent + alkali, or alkali salts alone. In soft water, a significant decrease in ash content is obtained when the washing agent is synthetic detergent + alkali. Little or no reduction is observed with soft water and soap + alkali or alkali salts alone. In no case does the addition of CMC result in an increase in the ash content. J.C.F.

**Decomposition of Sodium Chlorite in Bleaching Solutions**

H. Hefti

*Textil-Rund.*, 11, 82-85 (Feb. 1956)

The decomposition of sodium chlorite in acetic acid solutions of various pH values was studied at different

temperatures. The rate increases with falling pH and rising temp. On none of the laboratory-bleached cotton patterns could a change in the mean D.P. or carboxyl content be observed. S.R.C.

## PATENTS

**Dewaxing, Desizing, and Part Bleaching of Cotton Goods**

ICI

BP 747,481

Maximum absorbency can be obtained without kier boiling by a very brief treatment with an organic solvent followed by treatment with a peroxygen compound. The process can be made continuous and satisfactorily geared to continuous dyeing. Thus cropped and singed cotton drill was passed in open width through two vessels containing boiling trichloroethylene, the total time of immersion being 30 sec. It was then passed through boiling water for 10 sec. to flash off the solvent, and was then padded in an aqueous solution of sodium peroxide (10 g./litre), sodium silicate (5) and an octylated cresol-ethylene oxide condensate (0.5) to take up  $1\frac{1}{2}$  times its own weight of the liquor. The impregnated cloth was steamed for 1 min., rinsed successively in hot water, cold water, very dilute acetic acid and cold water, and finally dried. The treated cloth contained no starch, 0.31% wax, and had very good wettability. Similar cloth given a severe double kier boil had a wax content of 0.5-0.6% and contained traces of starch. C.O.C.

**Bleaching Vegetable Fibres with Peroxide**

Laporte Chemicals

BP 745,267

Bleaching is improved if it is carried out under pressure greater than atmospheric and if the peroxide is at  $> 100^\circ\text{C}$ . when it first meets the fibres. Thus cotton cheeses can be bleached at  $120^\circ\text{C}$ . in 30 min. with little chemical damage to the fibres. C.O.C.

Merits and Demerits of Continuous Processes of Bleaching and Dyeing and their Applicability to Indian Conditions (VIII p. 364)

**VIII—DYEING****"Optical Bleaching" in the Service of the Textile Industry**

P. Rochas

*Bull. Inst. Text. France*, (58), 27-47 (Feb. 1956)

The author outlines the physical principles underlying the action of fluorescent brightening agents, and discusses a number of points arising from their use—the objective evaluation of the whiteness of optically bleached fabrics; determination of the fluorescence colours of fluorescent brightening agents; the light fastness of these products, and their influence on the photochemical degradation of fabrics. In evaluating light fastness, due regard must be paid to the emission spectrum of the source, since fluorescent brightening agents are more sensitive to ultra-violet than to visible radiation. These agents do not increase the light-sensitivity of the common textile materials; in the case of nylon a slight protective action is apparent. J.C.F.

**Principles and Practice of Colour Stripping**

N. A. Matlin and W. C. Lindaly

*Amer. Dyestuff Rep.*, 45, P 168-P 172 (12 March 1956)

Factors influencing stripping action, e.g. diffusion through the solution, adsorption at the fibre surface, and diffusion within the fibre, are briefly discussed. Fibre permeability is of major importance and is responsible for the slow stripping rate of hydrophobic fibres, and it is essential to use an agent with good stability; experiments show that, in the time taken to strip e.g. cellulose acetate, hydrosulphite decomposes before the strip is complete, whereas zinc formaldehyde-sulphoxylate is satisfactory. Carriers may be of assistance, also helping the reaction products to desorb from the fibre. Scavenger systems (coacervates) are found to have certain disadvantages, though these are not necessarily inherent, and useful systems may exist. J.W.B.

**Effect of Penetration on Reflectance of Dyed Textile Fibres**

D. A. Garrett and R. H. Peters

*J. Textile Inst.*, 47, T 166-T 178 (March 1956)

The absorption and scatter coefficients  $K$  and  $S$ , as used by Atherton to define the reflectance of dyed textiles

(J.S.D.C., 71, 389 (1955)), are determined on the assumption that fibres of circular cross-section represent the absorbing and scattering elements through which the light passes. The ratio  $K/S$  is shown to be a function of fibre diameter, dye concentration, and extinction coefficient, and predicted and experimentally determined relations agree well. The use of the Atherton model is considered well justified, because the theoretical and observed relations between reflection changes and dye penetration correspond satisfactorily. In one example an increase in penetration from 10% to 100% is shown to have the same effect on reflectance as a 33% increase in dye concentration.

J.W.B.

#### Merits and Demerits of Continuous Processes of Bleaching and Dyeing and their Applicability to Industrial Conditions

M. M. Shah; K. G. Shah

*Textile Digest*, 16, 385-401; 403-421 (Oct.-Dec. 1955)

#### Spun-coloured Viscose Yarn

H. Palfreeman

*Canadian Textile J.*, 73, 57-58 (23 March 1956)

#### Hydrogen-bond Formation. IV—Hydrogen-bonding Properties of Water in Non-aqueous Solution and of Alcohols, Aldehydes, Carbohydrates, Ketones, Phenols, and Quinones in Aqueous and Non-aqueous Solutions

F. M. Arshid, C. H. Giles, and S. K. Jain

*J.C.S.*, 559-569 (March 1956)

The refractive index method for detecting complex formation in solution indicates that the OH group in alcohols is monofunctional in hydrogen-bond formation and, when present on vicinal C atoms in an aliphatic chain, forms weak five-membered chelate rings. In aldehydes, a variety of reactions demonstrate that the CHO group probably forms inter- and intra-molecular bonds through its H atom. In ketones, the CO group behaves bifunctionally, and the quinone O atom of quinones monofunctionally; ketones do not form inter-molecular complexes in benzene, ether, or water soln. In non-aq. solvents water appears to act either bifunctionally, probably by cross-linking, or monofunctionally in forming hydrogen-bond complexes with second solutions. In aq. soln. of carbohydrates, the open-chain form of glucose or cellobiose, in presence of such solutes as pyridine or amines, is stabilised by hydrogen-bond interaction of the CHO group; the OH groups are inactive through  $H_2O$  solvation, though active in ethylene glycol. In aq. soln., the OH groups are less reactive in the ring than in the open-chain forms of the simple carbohydrates. The bearing of these facts on the adsorption behaviour of cellulose is discussed.

H.H.H.

#### Influence of Modification of Wool on the Fastness of Dyeings to Aqueous Treatments

M. Van Overbeke, G. Decroix, and G. Mazingue

*Bull. Inst. Text. France*, (58), 49-56 (Feb. 1956)

The influence of (a) reduction by hydrosulphite, (b) acid treatment (simulating dyeing), (c) oxidation by alkaline  $H_2O_2$ , and (d) treatment with sulphuryl chloride in petroleum ether on (i) the affinity of wool for Neolan Red REG, and (ii) the fastness of the dyed samples to a standard wash test, has been examined. For modification by (a), (b), and (c), (i) increased and (ii) decreased progressively as the severity of the treatment was increased. Although a single (d) treatment resulted in an increase in (i) and a decrease in (ii), further treatments had little effect. The practical implications of the findings are discussed.

J.C.F.

#### Modifications to Wool. New Researches on the Ammoniacal Caustic Potash Swelling Test

C. Schirle, G. Gianola, and O. Meyer

*Bull. Inst. Text. France*, (58), 57-68 (Feb. 1956)

A simple method for mounting fibres parallel on a microscope slide is described. The fibres are treated with ammoniacal KOH (K.M.V. reagent), and the swelling  $G$  defined as—

$$G = \frac{\text{Diameter of fibre after swelling}}{\text{Initial diameter}}$$

is measured for 100 fibres. The cumulative frequency curve of the results is drawn, and the value of  $G$  ( $G$  50) corresponding to 50 fibres is obtained. The effect of various chemical treatments of different degrees of

severity on  $G$  50 has been examined. For treatment with acid, NaOH,  $H_2O_2$ , and thioglycolic acid,  $G$  50 first increases over the value for untreated wool and then decreases as the treatment is prolonged. Treatment with  $Na_2CO_3$  and degradation by light and heat, produce first a decrease and then an increase in  $G$  50. The use of the test to investigate the effect on wool of treatment in 0.1%  $Na_2CO_3$  soln. for the same time at various temperatures indicated that degradation is relatively slight up to 65°C., but increased markedly at higher temp., in agreement with the results of previous investigators. The test is a sensitive one and is suitable for assessing low degrees of modification.

J.C.F.

#### Variations in the Electrokinetic Potential of Acetate Rayon and Hydrophobic Substances in Solutions of Surface-active Compounds, with reference to Dyeing Theory

M. Palumbo and C. Festa

*Tintoria*, 52, 392-401 (Oct. 1955);*J. Textile Inst.*, 47, A 126 (March 1956)

Literature data on the  $\zeta$  potential of textile fibres are reviewed and experiments are reported to determine the adsorption of aqueous indigo suspensions on textile fibres in boiling solutions containing surface-active (cationic and anionic) substances. The surface-active agents were used in concentrations of 0.1-1% and the aqueous indigo suspension had a nitrogen content of 10-65%. The results, which are tabulated, show good agreement with the literature data referred to (14 references).

C.J.W.H.

#### Processes during the Dyeing of Synthetic Fibres with Disperse Dyes

B. Kramer

*Reyon Zellwolle Chemiefasern*, 607-609

(Sept.), 682-687 (Oct. 1955);

*J. Textile Inst.*, 47, A 127 (March 1956)

Investigation of the factors affecting the diffusion coefficient of dyes showed that the dipole moment is of fundamental importance for the dyeing properties of disperse dyes, hydrogen-bond formation being a specific case of dipole forces. Owing to their high dipole moment and the possibility of hydrogen-bond formation, azobenzene derivatives with terminal hydroxyl groups have satisfactory dyeing properties. In anthraquinone derivatives, the dipole moment can be increased by halogenation to obtain dyes that are suitable even for very hydrophobic fibres of high packing density (Latyl dyes for Terylene and Dacron). The carrier action of the dyes is reduced by the formation of intramolecular bonds. Resonance structures very easily react to different substrates, e.g. acetyl or amide groups, and the varying shades of several dyes on these fibres may be due to a rearrangement of the resonance structure. The dipole moment appears to be also of importance in dyeing polyacrylonitrile fibres with basic dyes (Deorlene-Atrazone).

C.J.W.H.

#### Physical Chemistry of the Dyeing of the new Textiles

J. Corbière

*Chim. et Ind.*, 75, 519-529 (March 1956)

PATENT

#### Urea as Dyeing Assistant

Courtaulds

BP 747,622

Textiles are treated with a dye in presence of urea and are then baked at a temperature between the m.p. of urea and 160°C. Thus woven cloth containing 67% viscose staple fibre and 33% regenerated protein staple fibre was padded to take up 110% of its weight of a solution of 0.4 g. Durazol Yellow GS (ICI), 0.4 g. Durazol Blue 2GNS, and 40 g. urea made up to 100 c.c. with water, the solution being at room temperature. It was then dried at 80°C. and baked for 10 min. at 140°C., washed with cold water for 10 min. and dried at 80°C. The dyeing was as fast as and more level than a similar one dyed in the normal manner. In another example a 100% viscose staple fibre cloth was padded with Solochrome Cyanine B (ICI) (1 g.), 32% Tw. basic chromium acetate (2 c.c.), and urea (50 g.) made up to 100 c.c. with water, dried at 80°C., baked for 4 min. at 145°C. and washed in cold water. The dried impregnated fabric was beige but turned blue during baking. The dyeing was fast to washing. Examples of the method used with acid dyes on wool, disperse dyes on Terylene, and solubilised vat dyes on acetate rayon are given.

C.O.C.

## IX—PRINTING

## Alician X Dyes in Textile Printing

W. Clarke

*Textil-Rund.*, 11, 136-144 (March 1956)

## PATENTS

## Photogravure Printing Plates or Rollers

R. Kaulen

BP 744,954

## Colour Correction in Photographic Colour Printing

Photo-Chemical Co.

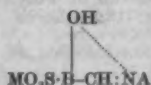
BP 748,183

## Yellow Schiff's Bases as Irreversibly Dischargeable Photographic Filter and Antihalation Dyes

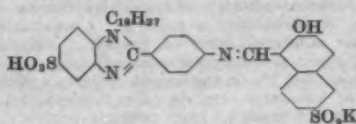
General Aniline

BP 747,632

Dyes of formula—



(M = H, alkali metal or a salt-forming group; B = radical of the benzene or naphthalene series; A = aromatic radical containing a sulpho or carboxy group), e.g.—



are used.

C.O.C.

## Transfers for Pottery and Opaque Vitreous Surfaces

Harrison &amp; Son (Hanley)

BP 745,713

The background of the design is first lithographed on to the paper, after which those portions of the design which are to be in relief are screen-printed on to the paper. When applied the transfers yield a design simulating hand-painted ware.

C.O.C.

## Xerographic Plate

Haloid Co.

BP 748,340

## X—SIZING AND FINISHING

## Sorption of Water by Cellulose Fibres at Low Humidities with special reference to Mercerised Cotton

J. B. Taylor

*J. Textile Inst.*, 47, T 147-T 153 (March 1956)

Sudan Sakel cotton is mercerised in NaOH soln. (280 g./litre) at room temp. for 10 min., X-ray examination indicating 90-95% mercerisation. Sorption data are obtained for regains up to 28.6 mg./g. at 25-26°C. Results are examined on the basis of Enderby's theory (*Trans. Faraday Soc.*, 51, 106 (1955)), in which the isotherm takes the form—

$$A = \frac{abP}{1 + bP} + cP$$

(A = amount absorbed; P = pressure; and a, b, c are constant at a given temperature). These requirements are satisfied by mercerised cotton and viscose rayon (Fibro) but not by cotton itself. Reasons for this are discussed.

J.W.B.

## Development of Shower- and Water-proofs

G. Dürig

*S.V.F. Fachorgan*, 11, 159-175 (April 1956)

After a discussion of early processes, modern proprietary products of various types are reviewed. The possible faults and testing methods are finally dealt with.

S.R.C.

## Photochemical Degradation of Polyamide Fibres

M. Schwemmer

*Textil-Rund.*, 11, 1-15 (Jan.), 70-82 (Feb.), 131-136 (March 1956)

The effects of long- and short-wave ultraviolet radiation on 6-, 6,6-, and 6,10-nylons were observed, and in the process a new method for conductimetric titration of carboxyl end-groups was developed, capable of use also with polyester fibres. It was found that chroming improved the light stability of polyamide fibres, trivalent chroming stabilising the peptide link.

S.R.C.

## Physical and Chemical Setting Tests

A. Leuckner

*Faserforsch. und Textiltech.*, 7, 124-131 (March 1956)

Some common setting processes and methods for testing them are reviewed. Their application to the setting of thermohydrofused Perlon is discussed. Solution behaviour, especially the time required to dissolve the polyamide filaments in sulphuric acid, gives the main information on the setting temperature.

W.R.M.

## PATENTS

## Antistatic Finish for Man-made Fibres

ICI

BP 747,827

Textiles containing synthetic polymers or cellulose acetate are treated with an organic polyisocyanate and a polyethenoxy compound containing > 1 OH. Thus a polyisocyanate (42 parts) (prepared by treating toluylene 2:4-diisocyanate (200) with glycerol (23) and diethylene glycol (21.2) in ethyl acetate (81.4) at > 90°C.) and a polyethylene glycol (20) of average mol.wt. 300 are dissolved in ethyl acetate (200). Nylon and Terylene immersed for 10 min. in the above solution, diluted with ethyl acetate (1 solution to 9 acetate for nylon, 1 solution to 2.3 acetate for Terylene), drained and baked for 50 min. at 120°C. received an excellent anti-static finish.

C.O.C.

## Mechanical Crimping of Nylon or other Yarn

Wildman &amp; Swartz

BP 747,320

## Coating Glass Filaments with Metal

B. F. Goodrich Co.

BP 747,453

## Carpets

William Kenyon &amp; Sons

BP 747,640

The pile is held in position by a synthetic polyamide whose fusing temperature is materially lower than that at which the other materials of the carpet are weakened. Thus a Wilton carpet has a pile of nylon and a weft of hemp previously impregnated with a warm alcoholic solution of nylon of low m.p. After weaving the fabric is heated to 200°C.

C.O.C.

## Compressive Shrinking of Woven Fabrics

Cluett Peabody &amp; Co.

BP 744,794

The moist cloth is stretched to the desired width and then over-shrunk warps by compressive shrinking while it is confined between a smooth hot surface and an elastic rubber surface. Much moisture is then flashed off as steam from the shrunk cloth and then the cloth is stretched in length so as to leave it with the desired shrinkage properties. This gives a smooth ironed finish on both faces of the cloth, shrinkage being as rapid as on a felt belt machine, and it enables closer tolerances to be obtained than with a rubber belt machine.

C.O.C.

## Flame-resistant Finish

Ciba

BP 747,014

The fabric is dried at raised temperature after being impregnated with an aqueous solution or suspension containing an ammonium or an organic nitrogenous base (b.p. or decomposes < 200°C.) salt of a phosphoric acid of O : P ratio less than that of orthophosphoric acid, an aminotriazine-formaldehyde condensate, and urea or a derivative thereof. Thus pyrophosphoric acid (20) is dissolved in water (80) and neutralised with ammonia. Urea (12), diacyandiamide (3) and a condensate (10) of unlimited water solubility and made from melamine (1 mol.) and HCHO (2-3 mol.) are then added. Cotton is impregnated with this solution, dried at 70°C., rinsed with cold water, and again dried. If two impregnations are given, a very good flame-resistant finish with no afterglow is obtained, the tensile strength and the handle of the material being unaffected.

C.O.C.

## Flame-resistant Finish

K. Quehl

BP 747,569

The material is impregnated with a solution or dispersion of a chlorinated polyvinyl chloride and one or more flame-resisting oxides of metals of groups IV and V. The polyvinyl chloride makes up < 6% of the compositions and its chlorine content must be < 60%.

C.O.C.

## Soil-resistant Treatment of Fabrics (VII p. 363)

Artificial Leather having a Polyvinyl Chloride Basis (XIII p. 366)

Slip Finish for Vinyl Resin Sheets or Coatings (XIII p. 366)

## XI—PAPER AND OTHER CELLULOSIC PRODUCTS

### Degraded Esparto Cellulose

G. O. Aspinall and W. B. Fordyce

*J.C.S.*, 683-687 (March 1956)

The preparation of a degraded esparto cellulose capable of peptisation into a colloidal soln. in  $H_2O$  is described. Methylation end-group assay shows it to have an average chain length of  $77 \pm 3$  glucose residues, a similar value being also obtained from periodate oxidation. The chemical accessibility of esparto cellulose, before and after degradation, is determined by acid hydrolysis and water sorption methods.

H.H.H.

### PATENT

#### Spirit Duplicating of Carbon Sheets

Ellams Duplicator Co.

BP 748,265

The coloured layer is coated with a layer containing an ingredient which forms a water-insoluble compound with the dye in the coloured layer, e.g. tannic acid where a basic dye is used. This enables the paper to be handled without staining the fingers.

C.O.C.

#### Static Electricity in the Textile and Paper Industries (VI p. 362)

Effect of Degree of Substitution on the Fractionation of Cellulose Nitrates (XIV p. 367)

## XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

### Swelling of Protein Molecules in Solution

T. L. Hill

*J. Phys. Chem.*, 60, 358-361 (March 1956)

The suggestion of Yang and Foster that serum albumin swells at low pH because of an  $\alpha \rightarrow \beta$  transformation is examined theoretically, and the mechanism suggested is found to be a reasonable possibility. The theory, however, predicts a salt effect opposite in sign to that found experimentally.

W.R.M.

### PATENTS

#### Tanning Agents and Tanning

FBY

BP 747,092

Water-soluble tanning agents are obtained by condensing an aldehyde with a salt of an amine or of ammonia and a polyhydric phenol which may be partly replaced by a monohydric phenol ( $> 1$  mol. salt to 1 mol. phenol). They have excellent tanning properties even in weakly acid liquors.

BP 747,093

An  $\alpha\beta$ -unsaturated aldehyde (1 mol.), e.g. acrolein or crotonaldehyde, is condensed with resorcinol (2 mol.) in concentrated aqueous solution in presence of a mineral acid as catalyst. The insoluble product is treated with a sulphite and/or polyhydric phenol or a salt of an aromatic amine ( $> 1$  mol. per mol. of aldehyde) preferably at  $> 100^\circ\text{C}$ . under pressure. The three types of water-soluble products obtained are all tanning agents.

BP 747,094

The amphoteric condensates obtained from an aldehyde, 1-10 mol. of a polyvalent phenol and 1 mol. of an ammonium or amine salt are water-soluble tanning agents. They are used so that the pH of the bath increases towards the end of tanning.

C.O.C.

#### Dicyandiamide-Formaldehyde Resins for treating Leather

Jacques Wolf & Co.

BP 747,183

A resin stable in aqueous solution is obtained by heating an aqueous solution of 3-6-5-0 mol.  $HCHO$ , 1 mol. dicyandiamide and 0-3-1-0 mol. boric acid until a product is obtained which is soluble in water on extreme dilution and which, after adding enough of a water-soluble anionic surface-active sulphonate to dissolve any precipitate first formed on adding the sulphonate, gives a precipitate with aluminium sulphate. Application of the resulting solution to leather, particularly chrome-tanned leather, which if not anionic should be rendered so by treatment with an anionic agent, results in increased weight or thickness of the leather, improved whiteness and finer and

improved overall "break" (resistance to sharp creasing and attendant separation of fibres when the leather is bent).

C.O.C.

### Body Colour or Top Finish for Leather

Chemische Fabrik Grünau

BP 747,769

Incorporation of a condensate of soluble protein and/or protein decomposition products with higher fatty acids, resin acids, naphthenic acids and/or higher aliphatic or aliphatic-aromatic sulphonic acids yields products which have good adhesion to leather, glaze satisfactorily, and have good resistance to water and wet rubbing.

C.O.C.

## XIII—RUBBER; RESINS; PLASTICS

### Thermal Decomposition of Polytetrafluoroethylene in Various Gaseous Atmospheres

L. A. Wall and J. D. Michaelsen

*Bur. Stand. J. Res.*, 56, 27-34 (Jan. 1956);  
Research Paper 2644

### PATENTS

#### Artificial Leather having a Polyvinyl Chloride Basis

Deutsche Gold- und Silber-Scheideanstalt

BP 748,046

A product permeable to air and water vapour but impermeable to water is obtained by mixing polyvinyl chloride, a plasticiser, a pigment, a swellable macromolecular substance inert to polyvinyl chloride and a swelling agent for the macromolecular substance. There must be at least 3% on the weight of the mix of the macromolecular substance, e.g. starch or benzylcellulose. The swelling agent must be inert towards polyvinyl chloride and must volatilise at the temperature at which polyvinyl chloride gels. The mix is then formed into a sheet or coated and heated to gel the polyvinyl chloride.

C.O.C.

#### Slip Finish for Vinyl Resin Sheets or Coatings

United States Rubber Co.

BP 747,229

The vinyl resin sheet or coated fabric is treated with a volatile organic solvent containing resinous polyethyl methacrylate, resinous polymethyl methacrylate, polyvinyl chloride or a copolymer of vinyl chloride and vinyl acetate containing  $> 50\%$  of vinyl chloride, and either a silicone fluid or a ketone-soluble silicone rubber.

C.O.C.

#### Coloured Thermoplastic Masses for Injection Moulding

Chemische Werke Hüls

BP 747,486

Coloured thermoplastic polymers of compounds containing a carbon double linkage for injection moulding are obtained by treating a granular thermoplastic polymer with a dispersion of a non-oil-soluble pigment in a solution of a resin. The solvent used must not be a swelling agent for the polymer and must be readily volatile.

C.O.C.

#### Improving the Physical Properties of Polymers by High-energy Irradiation

United Kingdom Atomic Energy Authority

BP 747,478

Polymers of mol. wt.  $> 25,000$  when given brief exposure to high-energy radiation, e.g.  $\gamma$ - or X-rays, have better physical properties than those of lower average mol. wt. which have been similarly exposed for longer periods. In particular tensile strength at  $> 120^\circ\text{C}$ . is increased.

C.O.C.

#### Printed Rubber Sheet or the like

Woven Hose & Rubber Co.

USP 2,712,704

The design is printed on the sheet which is then stretched in one or both directions and set while stretched. This blurs the outlines of the design, establishes gradations between and modifies the colours. If the sheet is stretched beyond the tensile limit of the ink then a mosaic effect is obtained.

C.O.C.

#### Decorated Synthetic Resin Surfaces

Röhm & Haas

BP 744,863

A liquid containing pigments is polymerised in a chamber having at least one wall recessed so as to cause the surface of the solid polymer to have raised portions. The amount of pigment used is such that it settles only in the recesses. This yields articles having a surface decorated with raised portions which contrast in colour and/or light permeability with the body of the article.

C.O.C.

#### Improving the Dyeing Properties of Synthetic Polypeptides (VI p. 363)

# XIV—ANALYSIS; TESTING; APPARATUS

## Simultaneous Microanalytical Determination of Carbon, Hydrogen, and Nitrogen

W. Schöniger

*Helv. Chim. Acta*, **39**, 650-652 (March 1956)

Organic comp. are burnt in presence of CuO in a closed and evacuated ( $10^{-3}$  mm. Hg) combustion tube containing CuO, Ag, and Ni. Only CO<sub>2</sub>, H<sub>2</sub>O, and N<sub>2</sub> are formed. The first two are condensed in traps by means of liquid air and solid CO<sub>2</sub>-acetone respectively, the residual pressure corresponding to the N content. N<sub>2</sub> is sucked off, the system again isolated, and first the CO<sub>2</sub> and then the H<sub>2</sub>O released by warming, the resulting pressures leading separately to the C and H contents. One weighing only and calibration of the apparatus are required. The ratio C:H:N can be obtained without any weighing. H.E.N.

## Polarographic Behaviour of Sodium Dithionite (Hydrosulphite)

V. Čornák

*Chemické Zvesti*, **8**, (10), 714-720 (1955)

The polarographic behaviour of sodium dithionite in neutral and alkaline medium is determined by the reaction—



i.e. decomposition of a dimer into two monomer molecules. Theoretical and practical results agreed well, confirming the postulated equilibrium—



in solutions and permitting calculation of the ratio of the rate constant  $k$  for decomposition of the dimer and the equilibrium constant  $K = [D]/[M]^2$ . A reaction scheme is put forward which can explain also the polarographic behaviour of dithionite in acid solutions and the mechanism of the electrode reactions of sulphurous acid. C.J.W.H.

## Wool Damage Test with Cuprammonium Solution (RW Method)

G. Nitschke

*Faserforsch. und Textiltech.*, **7**, 119-124 (March 1956)

Damage to wool can be estimated by treatment with cuprammonium solution of known concentration and subsequent iodometric determination of the copper taken up by the wool. An improved method is described. Attempts are made to find the nature of the bond between copper and wool and its dependence on the type of damage. Alkali solubilities and physical properties of the wool are determined for comparison. Acid and oxidative damage and the influence of treatment with formalin solutions are also considered. W.R.M.

## Ishihara Test and Defects in Colour Vision

P. Cavanagh

*Occupational Psychology*, **29**, 1 (1955)

*Nature*, **176**, 816 (29 Oct. 1955)

An account of an investigation originally undertaken for the guidance of psychologists working in Industrial Rehabilitation Units during the Second World War. The author discusses the incidence and the types of defective colour vision, its congenital nature, and its occupational implications, with a detailed study of the test, its limitations and value, and the technique for reducing error. C.J.W.H.

## Determination of pH and Errors in Colorimetric Measurement in Textile Finishing

C. Adrian

*Industria textila* (Bucharest), **6**, 65-71 (1955)

*Chem. Abs.*, **49**, (17), 12011 (1955)

*J. Textile Inst.*, **47**, A 36 (Jan. 1956)

The presence of proteins, colloids, etc. leads to erroneous results when certain indicators are used in textile-finishing control. The effect of these factors is shown with reference to a number of indicators. A review is made of pH-measuring methods. C.J.W.H.

## Damage caused by Retained Chlorine

AATCC Committee on Damage caused by Retained Chlorine

*Amer. Dyestuff Rep.*, **45**, P 135 (27 Feb. 1956)

The Committee's position is summarised in view of difficulties which have arisen in evaluating damage and in obtaining a reproducible test method, and a list of important factors is given. J.W.B.

## Continuous Moisture Measurement on Wide, High-speed Paper Machines

K. Fetter

*Svensk Papperstidning*, **59**, 61-65 (31 Jan. 1956)

The Supertest moisture recorder, based on the dielectric measurement of the moisture content of paper, is described. The sensing device, incorporating a condenser passing over the paper surface, is mounted on a double-channel beam on the machine to allow it to travel freely across the width of the machine. Two recorders in the central instrument panel register continuously the average moisture content of the web and the change of moisture content across the web. The sensing head is removed automatically from the path of the web in case of breaks. A special moisture-control device intended to work in conjunction with the Supertest is under development. K.W.

## Effect of Degree of Substitution on the Fractionation of Cellulose Nitrates

C. F. Bennett and T. E. Timell

*Svensk Papperstidning*, **59**, 73-79 (15 Feb. 1956)

Cellulose nitrates, with N contents ranging from 13.62% to 14.14%, prepared without degradation from acetate-grade cotton linters, were fractionally pptd. in one stage with acetone and either water or hexane. The chain-length distributions of the partly substituted nitrates were found to be almost identical to that of the completely nitrated product; hence, provided that the average N content is  $< 13.5\%$ , its influence on the results obtained in one-stage fractional pptn. is negligible. The chain-length distributions obtained with hexane and with water were similar, but the latter allowed a larger number of fractions to be isolated, so that the recently described suggested standard fractionation method (Bennett and Timell, *ibid.*, **58**, 281 (1955)) appears to be the best available at present. K.W.

## Independent Measurement of the Amorphous Content of Polymers

R. G. J. Miller and H. A. Willis

*J. Polymer Sci.*, **19**, 485-494 (March 1956)

An infrared spectroscopic method is proposed for the determination of the amorphous content of polymers. The method depends on the measurement of the intensity of pure amorphous bands. The application of the method to some common polymers is described and the results are compared with those obtained from X-ray and density measurements. W.R.M.

## Esterification of Peptides and its Significance in Determinations by Reduction of the Amino-acid Residue at the Carboxyl End

H. Hörmann, W. Grassmann, E. Wünsch, and H. Preller

*Chem. Ber.*, **89**, 933-945 (April 1956)

## Burning Test on Flame-resistant Tight Textiles

using Vertically Stretched Samples

DIN 53,906

*Faserforsch. und Textiltech.*, **7**, 179-181 (April 1956)

The sample is placed vertically in a flame of fixed height in a closed box. The time taken to extinguish the flame is noted. A table is given relating the time to the weight of the material in g./m.<sup>2</sup>. Glow time also is recorded. A tearing test to follow the burning test is given. Methods of selecting the sample and apparatus are described and illustrated. W.R.M.

## Burning Test on Flame-resistant Open Textiles using Horizontally Stretched Samples

DIN 53,907

*Faserforsch. und Textiltech.*, **7**, 181-182 (April 1956)

The sample is stretched on a frame and placed in a draught-free box. An alcohol burner is placed under the centre of the sample, and 0.3 ml. of alcohol burnt. The time from the extinction of the alcohol flame to extinction of the flame of the sample is determined. Glow time is also recorded. Selection of the sample and the frame are described and illustrated. W.R.M.

## The Opacimeter—An Essential Tool for Paper and Print

Technical Section, British Paper & Board Makers' Assocn.

*Proc. Tech. Sectn. British Paper & Board Makers' Assocn.*, **36**, 617-638 (Dec. 1955)

As a supplement to B.P.B.M.A. Testing Method PT 24: pm 1955, *The Printing Opacity of Paper*, three types of commercial opacimeter are described, and causes

of errors commonly encountered in their use are explained. Results of comparative tests and their statistical examination are summarised. Finally, the conversion of printing opacity to contrast ratio or vice versa is discussed.

K.W.

#### Determination of the pH Value of Paper

J. Grant

*Proc. Tech. Sectn. British Paper & Board Makers' Assocn.*,  
36, 473-497 (Dec. 1955)

Existing proposed standard methods of extracting paper for pH and similar evaluations are listed and discussed with special reference to the technique of preparing the extract of paper. The final draft method PT 22 : pm 1955, which includes both cold and hot extraction methods, is presented with comments.

K.W.

#### Determination of Water-soluble Chlorides and Sulphates in Paper

J. R. Ainscough and F. Bridge

*Proc. Tech. Sectn. British Paper & Board Makers' Assocn.*,  
36, 449-471 (Dec. 1955)

Chlorides and sulphates increase the electrical conductivity of paper under humid conditions, thus favouring the corrosion of metals in contact with damp paper. Potentiometric and nephelometric methods are described that have been found suitable for the determination of chlorides and sulphates respectively. Statistically designed experiments have been carried out to study the effect of variations in extraction conditions on the results obtained.

K.W.

#### PATENTS

##### Measuring Moisture Content

Industrial Machinery Co.

BP 745,917

A radiation gauge, e.g. a  $\beta$ -ray gauge, measures the weight per unit area of the material while the material is moving. Another gauge measures one or more of the electrical characteristics of the material affected by moisture, e.g. dielectric properties. The electrical output of the two gauges is combined to measure the moisture content of the material and/or control the moisture content.

C.O.C.

##### Paint-testing Machine

H. R. Harlan

USP 2,712,235

A machine which measures the brushability as distinguished from the viscosity of paints, etc.

C.O.C.

Soil-resistant Treatment of Fabrics (VII p. 363)

Surface Properties of Carboxymethylcellulose (VII p. 363)  
"Optical Bleaching" in the Service of the Textile Industry (VIII p. 363)

Effect of Penetration on Reflectance of Dyed Textile Fibres (VIII p. 363)

Modifications to Wool. New Researches on the Ammoniacal Caustic Potash Swelling Test (VIII p. 364)

Development of Shower- and Water-proofs (X p. 365)

Photochemical Degradation of Polyamide Fibres (X p. 365)

Physical and Chemical Setting Tests (X p. 365)

#### XV—MISCELLANEOUS

##### Metal Colouring by Electrodeposition of Thin Interference Film. I—Determination of Optimum Colouring Conditions

M. Kikuchi

*Rept. Govt. Chem. Ind. Research Inst. Tokyo*,  
51, 27-31 (Jan. 1956)

The optimum conditions were determined for metal colouring by electrodeposition of a thin film which displays interference colours. A copper plate was polished, cleaned, and immersed as the cathode in the complex solution consisting of copper sulphate, Rochelle salt, and caustic soda, their concn. being 0.1, 0.1, and 2 M, respectively. The anode was a carbon or a platinum plate, and the electrolysis was done at room temp. (25-30°C. was suitable). On the current-voltage curve, two stages were found for

the electrolytic reactions. The thin film displaying the interference colours was formed at the first reaction stage corresponding to the lower part of the curve. The limiting current density for the first reaction was about 60 ma./dm.<sup>2</sup>. The colours developed were orange, red, yellow, and golden yellow. The first-order colour sequence was finished in about 5 min. After three repetitions of the developed colour sequence, the permanent red film was formed on the cathode. At the second reaction stage, metallic copper was deposited.

AUTHOR

##### Ultraviolet-absorbing Filters

H. Ikeda and K. Toba

*Rept. Govt. Chem. Ind. Research Inst. Tokyo*,  
51, 65-73 (Feb. 1956)

As absorbents for ultraviolet, Tartrazine, ultrazine, R acid, G acid, and aesculin are used traditionally, but they have the defects of transmittance in the near-ultraviolet region, absorption of blue light, fluorescence, or instability to light. The aim was to make ultraviolet-absorbing filters which are colourless, non-fluorescent, clear, stable to light, and having a sharp cut-off. The light transmittance of flavone, flavanone, chalcone, and coumarin were measured at an absorbent concentration of 3 g./m.<sup>3</sup> in alcohol or benzene. Interesting relations were found between absorption curves and chemical constitution: the absorption edges of flavones shift to longer wavelengths with an increase in the number of OH groups, and to shorter wavelengths with a decrease. The flavone and coumarin groups have sharp absorption curves, but the chalcone and flavanone groups have rather flat curves. The flavone group is the best absorbent, because, besides having a sharp cut-off for the ultraviolet, it is non-fluorescent and is fast to light. With these agents several kinds of filters were made.

AUTHORS

#### PATENTS

##### Lead Coating of Ferrous Metals

Steel Ceilings

BP 747,733

The metal surface is treated with an aqueous solution of a Pb salt of an aliphatic polybasic hydroxy acid, e.g. malic, tartaric, citric, maleic or diglycolic acids, at pH < 7 and Pb ion concentration of 3-50 g. per litre.

BP 747,734

Build-up of iron in the solution is limited by adding an ammonium or alkali-metal fluoride or bifluoride, which precipitates the ferric iron without affecting the lead or other divalent metal present.

BP 747,735

An aqueous solution of a Pb salt of an aliphatic monobasic hydroxy acid, e.g. glycolic, lactic and gluconic acids, is used at pH < 7 and Pb ion concentration of 50-300 g. per litre.

C.O.C.

##### Colouring of Metal-coated Sweets

J. Harradine &amp; Sons

BP 749,426

Silver-coated sweets used for decorating cakes are coated with a colouring material and shellac or other transparent film-forming material dissolved in a highly volatile solvent.

C.O.C.

##### Sulphide Coating of Stainless Steel

Parker Rust Proof Co.

USP 2,715,083

The metal is treated with an aqueous solution containing a sulphur compound e.g. Na<sub>2</sub>SO<sub>3</sub>, the fluoride ion e.g. from KF and enough acid e.g. nitric acid to keep the pH < 0.5.

C.O.C.

##### Methylene Blue as Polymerisation Inhibitor for Diacetylene

Monsanto

USP 2,715,101

##### Imitation Down

U.S. Secretary of the Army

USP 2,713,547

A monofilament having an irregularly curled, twisted form is coated with an adhesive, feathers or disintegrated feathers are blown against it, after which it is cut into short lengths. Batts made from the product have high filling power and can be substituted advantageously for the duck feather and down mixture often used in sleeping bags, etc.

C.O.C.



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All inquiries relating to Advertisements should be addressed to THE GENERAL SECRETARY, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE.

Replies may be addressed Box —, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE, where all communications relating to these Advertisements are treated in strict confidence.

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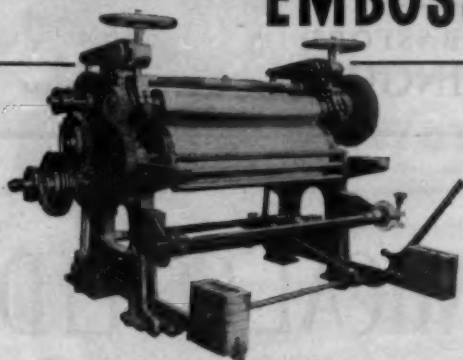
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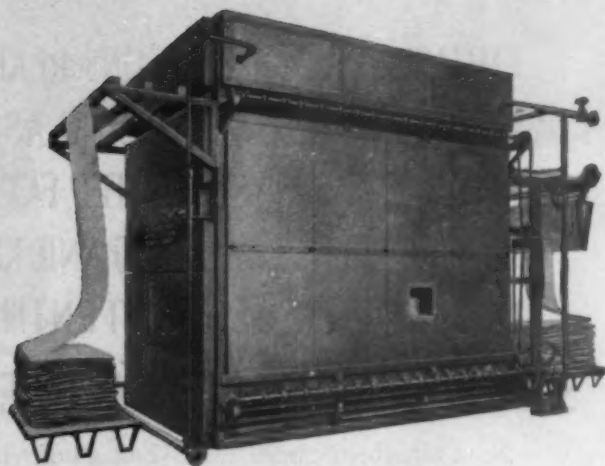
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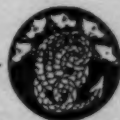
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**1931**

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1931 was also the year in which Laporte successfully introduced an electrolytic method of manufacturing Hydrogen Peroxide.

Laporte have held the lead since 1888 in the manufacture of this important product, application of which now varies from bleaching textiles to a compact source of concentrated energy for driving submarines and rockets.

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